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BACKSTREAMING AND 'INERT ATMOSPHERE'

VACUUM as an inert atmosphere, discussed in our last issue, was considered as a rarefied atmosphere reducing the risk of contamination by the constituents of that atmosphere, while manipulating or processing a component or material.

In the early days of vacuum technology, the mercury vapour pump with a trap refrigerated to a very low temperature was virtually the only practical means of obtaining low pressures. The advent of the oil vapour pump was considered a great step forward because low ultimate pressures could be obtained without liquid-gas cooling, with consequent relief from the inconvenience of maintaining the traps operative. At that stage of development the small quantities of organic vapour introduced into the vacuum system from backstreaming, etc., were acceptable for most processes.

Ever-rising standards of 'cleanliness' have, however, produced new problems in the use of oil diffusion pumps, and in some very exacting applications it has been necessary to revert to mercury vapour pumps, or adopt other techniques, as mentioned in the previous issue. Because, however, of the ease of operation of oil vapour pumps, major efforts have been made to retain the oil vapour pump over as wide a range of applications as possible.

The degree of organic contamination arising from an oil vapour pump can be reduced in various ways. Pumping fluids are now available with vapour pressures many times lower than those originally employed, and the development of better fluids is actively pursued. Fractionating techniques introduced to ensure the use of the most phlegmatic constituents for the high vacuum stage have advanced to a considerable degree. Also, oil pumps can be made to eject the least desirable fluid components. Thus it is in fact possible for a good modern oil vapour pump, together with a well-designed water-cooled baffle, to be satisfactorily used where the early types would be quite unsuitable.

The function of such a baffle is to intercept and condense vapour molecules migrating back into the system in excess of those which would normally arise by evaporation from the cooled pump walls. It is this excess, defined as backstreaming, which can often increase the vapour pressure in the system above the condensation point at system temperature, so that continuous condensation occurs on surfaces within the system, and serious surface contamination results.

If the amount of backstreaming can be reduced at source, then a simpler and less speed-restrictive baffle may be provided, with nevertheless, more efficient baffling. There is the additional advantage that the surfaces of the baffle remain clean for a longer period.

The results of the investigation presented in this issue show that backstreaming can be controlled, and hence the oil vapour pump with all its simplicity of operation and construction, may continue to play its effective part, in spite of the claims of new pumps such as the getter pump and the mechanical booster pump to overcome the vapour contamination problem.

It will be apparent that the measurement of backstreaming rates is a matter of considerable finesse and, as a start has been made by one or two manufacturers to quote the backstreaming rates of their pumps, it is extremely important in the interest of the user that an internationally agreed procedure of measurement is established.

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Mr. Power was born in 1918 at Newcastle-on-Tyne. He received his general education at St. John's School, Leatherhead, and joined St. John's College, Cambridge, in 1937. He took his Mechanical Sciences Tripos Course and became a Bachelor of Arts in 1940. Mr. Power proceeded to his M.A. in 1948.

In 1946 he joined the Research Laboratories of Edwards High Vacuum Ltd., and soon became the Senior Assistant in the Pump Section. He was appointed Head of the Section in 1951.

In his capacity as a pump specialist, Mr. Power had a large share in the theoretical and experimental work spent on the development of the rotary and, in particular, the vapour pumps put on the market by that Company during the last ten years. The design of the vapour booster pumps evolved by the team under his leadership represented an especially noteworthy success as these pumps differed in many important features from those made available during the same period in other European countries.

Mr. Power began to publish on his work in 1951. Two articles for which he was a co-author with other of his colleagues—'Investigations into the Ultimate Pressures of Diffusion Pumps Designed to Purify their Working Fluids,' and 'An Assessment of Some Working Fluids for Diffusion Pumps'—published in this Journal, made notable contributions to vacuum engineering literature, and his contribution to this issue is an indication of the important developments that still lie ahead of the vapour pump.

DEREK J. CRAWLEY, B.Sc., was born in London in 1925, and received his general education at Alleyn's School, Dulwich. After serving in the Forces from 1944 to 1947 he joined the scientific staff of the Fuel Research Station, D.S.I.R. He entered University College, Leicester, in 1949 and graduated in 1951. He then joined Edwards High Vacuum Ltd. and is now a member of the team led by Mr. Power. Mr. Crawley has been particularly concerned and interested in vapour pumps, in the development progress of which he has made significant contributions.

SOURCES, MEASUREMENT AND CONTROL OF BACKSTREAMING

in oil vapour vacuum pumps

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Summary

IN ALL VAPOUR DIFFUSION PUMPS working fluid vapour continuously backstreams through the pump inlet to an amount depending on the design and method of operating the pump, with the result that the vapour pressure in the pumped system is increased beyond that corresponding to the temperature of the pump-cooling water, and undesirable condensation of fluid in the system may ensue. The sources of the backstreaming vapour are studied and methods of control discussed which do not adversely affect the pump performance. A critical review is made of various methods of measuring backstreaming, and the technique adopted by the authors is described. The lip of the top jet nozzle is shown to be an important source of backstreaming, and a qualitative explanation for this is put forward. Two new control devices are then described: One, a cooled 'guard ring' around the nozzle lip which intercepts the backstreaming vapour at source, and the other, a nozzle with a controlled temperature gradient along its wall which is intended to condense the randomly moving outer layer of the vapour jet from which the backstreaming molecules originate. This principle is actually contrary to recognized good practice.

Sommaire

DANS TOUTES LES POMPES A DIFFUSION la vapeur du fluide employé remonte continuellement au travers de l'entrée de la pompe, en quantité dépendant de la construction et de l'utilisation de la pompe; en fait, la tension de vapeur dans le système est plus élevé qu'elle ne serait à la température de l'eau de refroidissement de la pompe, et une condensation indésirable des fluides dans le système peut en resulter. Les sources de remontée de vapeur sont étudiées, et on discute les méthodes de controle qui n'affectent pas les performances de la pompe. On fait un examen critique des différentes méthodes de mesure du phénomène de remonteé, et la technique adoptée pa les auteurs est décrite. On démontre que le bord de l'éjecteur de l étage supérieur est une source importante de remontée, et une explication qualitative est avancée. Deux nouveaux dispositifs de controle sont décrits; l'un est un anneau de garde refroidi, fixé autour du bord de l'éjecteur, interceptant la remontée de vapeur à sa source, l'autre un éjecteur ayant sa paroi à gradient de température controllé ce qui doit condenser la zone externe du jet de vapeur d'ou les molécules formant la remontée de vapeur proviennent. Le principe est en effet contraire à la pratique normale.

INTRODUCTION

IN THE ABSENCE OF refrigerated or sorption traps, the ultimate vacuum obtainable by an oil diffusion pump cannot be lower than the saturation pressure at wall temperature of the condensate on the cooled pump wall around the first jet of the pump, and will in general be higher due to pressure components from other sources. Chief among these components are:

(*a*) Permanent gases and light vapours arising largely from pump fluid decomposition in the boiler. These components can be kept fairly small compared with condensate vapour components in a well-designed pump. The work now being reported is in no way concerned with them, and they will not be discussed further.

(*b*) Pumping fluid vapour in excess of that corresponding to saturation vapour pressure at cold wall temperature and arising mainly because some

* MS. received February, 1957.

of the vapour molecules emitted from the first stage jet migrate backwards and issue from the mouth of the pump without ever reaching the cooled pump wall, or possibly after a collision with the wall during which accommodation to

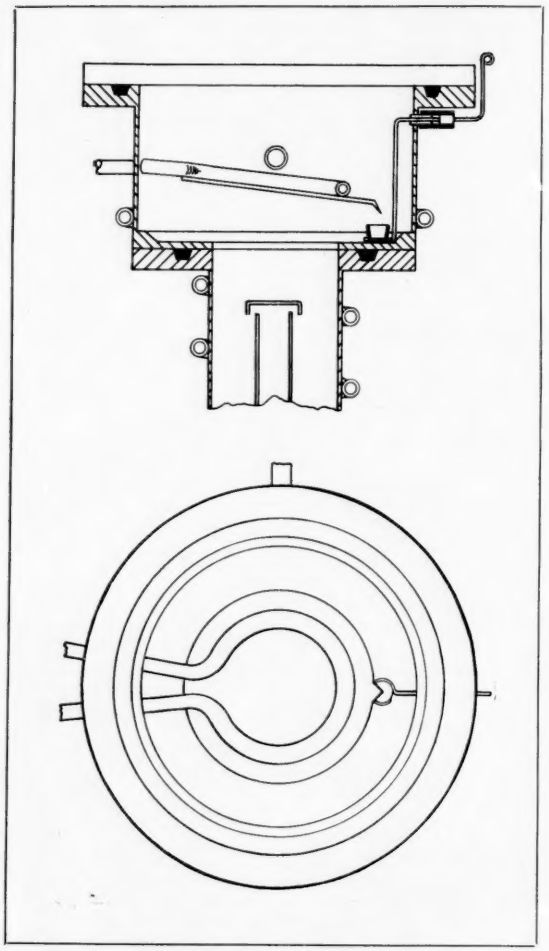

Fig. 1*a*. Measuring apparatus using water-cooled collector for small pumps.

wall temperature was incomplete. This excess of vapour moving backwards from the pump mouth is generally referred to as 'backstreaming' vapour. It is becoming increasingly important to suppress backstreaming and this problem is the central topic to be discussed.

If there were no backstreaming, a vapour pressure corresponding to saturation pressure at pump cold-wall temperature would be present nevertheless in the pumped system (in the absence of traps), but there would be no condensation on surfaces in the system because the cooled pump walls are usually colder than the system surfaces. The extra vapour contributed by backstreaming can however so increase the vapour pressure in the system that the saturation point is exceeded, condensation on surfaces within the system is continuous and there is a steady net transfer of pumping fluid into the system.

There is an increasing number of vacuum applications where such surface contamination cannot be tolerated. Examples are the deposition of certain coatings by the vacuum evaporation technique, some classes of work involving the use of emitting filaments or secondary emission surfaces, apparatus where targets are bombarded by electron or ionic beams and target contamination cannot be tolerated, and apparatus where high-voltage insulators must retain clean uncontaminated surfaces.

Certain of the above applications may be so exacting in their requirements for a 'clean' vacuum, free from contamination by organic vapour, that even the legitimate vapour pressure corresponding to the pump wall temperature, or the vapours arising from organic materials used in the system being pumped, may be sufficient to cause troublesome contamination[1] and in such cases it may be quite unjust to condemn the pump for backstreaming excessively. In some cases the solution of the contamination problem may involve the use of refrigerated baffles or special traps, or the use of mercury pumps with liquid air traps, or the complete elimination of organic materials from the whole system.

The standard method of controlling backstreaming is to provide between pump and system a water-cooled baffle so designed that every backstreaming molecule which travels straight must collide with it. Cooled by the same cooling water as that passing through the pump, the baffle is intended to ensure that the pressure on the system side of it is reduced to saturation

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pressure at pump-cooling water temperature and that all excess backstreaming vapour is condensed out and returned to the pump. Vapour molecules may however collide with each other and not in fact travel in straight lines; moreover, they may not always accommodate completely to baffle temperature after a single collision, so that no practical baffle can be completely efficient. Baffling efficiency can be increased by designing a more restrictive baffle such, for example, that at least two collisions are inevitable for a straight travelling molecule, but such a baffle would severely throttle the speed of the pump for gas, and most practical baffles must be designed to provide a fair compromise between high baffling efficiency and minimum throttling of pump speed.

The consequences of employing a baffle which is not completely efficient are obviously less serious if the initial backstreaming rate is itself very small, for any reduction that can be made in the unbaffled backstreaming rate is likely to result in at least a proportional reduction in residual backstreaming after baffling. So it is clearly desirable to try to reduce backstreaming even if a baffle will be used in any case. In the case when a refrigerated baffle is employed which freezes the pumping fluid it collects, a reduced backstreaming rate will increase the time it is possible to run before the pump has been robbed of too much operating fluid.

The authors have been concerned with the study of backstreaming for several years and some of their early measurements have already been reported.[2] The results submitted in the present article have been accumulated during a great many investigations into various aspects of pump design and do not represent the result of a single investigation.

MEASUREMENT OF BACKSTREAMING

Discussion of Techniques

Backstreaming is usually measured by providing a collector plate of some sort above the mouth of the pump and by weighing the amount of pumping fluid collected on it in a measured time. Any such method should however discriminate between the vapour inevitably present above the pump mouth, corresponding to saturation pressure at pump wall temperature, and the extra vapour issuing from the pump mouth which we have defined as 'backstreaming'.

If the collector plate were chilled, it would condense vapour of both categories and liquid from the pump walls would actually be distilled over onto the collector. A most erroneously high value for the backstreaming rate would be indicated. On the other hand a lightly supported collector plate without any definite temperature control would be likely to acquire a temperature somewhat higher than the cold pump walls since, even if the test header walls are water-cooled,

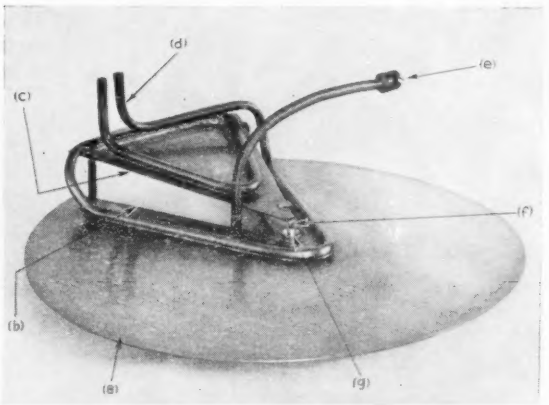


Fig. 1*b*. Measuring apparatus using water-cooled collector for large pumps. *Legend:* (*a*) Copper disc covering pump mouth inside test dome; (*b*) Cut-away sector of disc; (*c*) Copper collector plate overlapping cut-away sector; (*d*) Cooling coil soldered to disc and plate; (*e*) Flexible control for moving cup; (*f*) Extended tongue at lowest part of collector; (*g*) Collecting cup.

the collector must necessarily be exposed to radiation from the hot jet assembly and vapour stream of the pump. Re-evaporation of condensate from such a collector would therefore be at a higher rate than that appropriate to equilibrium conditions at pump-wall temperature, a proportion of the captured 'backstreaming' fluid would escape to condense on colder parts of the system and an erroneously low and somewhat random value for the backstreaming rate would be recorded.

After some initial experiments it appeared that the

soundest method of measurement was to provide a water-cooled collector in a water-cooled test header and to circulate through both of them the pump-cooling water on its way to the pump-water inlet. Since test header walls, pump walls above the first stage and collector are now kept at sensibly the same temperature, there is no significant *net* distillation of fluid from one to the other during a test. Only the genuine backstreaming excess of vapour is acquired by the collector as condensate and none is lost by re-evaporation.

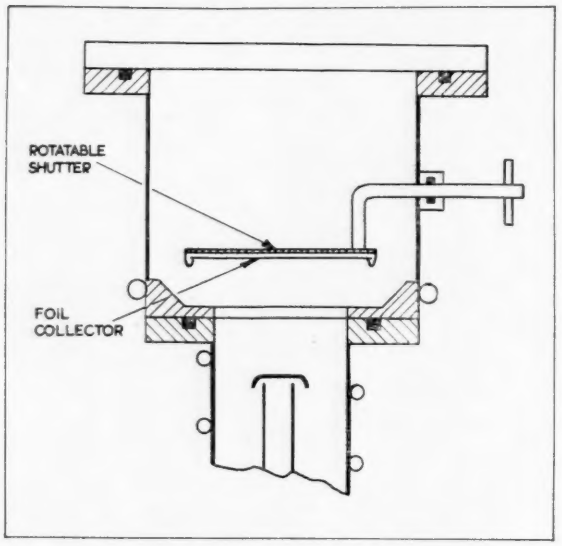


Fig. 2. Measuring apparatus using uncooled paper or foil collector.

It is necessary, in addition, to ensure that substantially all the backstreaming vapour emerging from the pump mouth is caught by the collector, so the collector should be either in the plane of the pump mouth and covering the whole of it or, if above the pump mouth, should well overlap it all round. A collector set high in a test header of the same bore as the pump can obviously receive only a proportion of the backstreaming vapour.

On larger pumps it is convenient to collect only over a portion of the total mouth area. In this case the collector should be a complete sector of the normal disc. The backstreaming flux density, though varying along a radius, may be taken as having a uniform distribution about the pump axis so that measurement over a sector allows the total to be obtained by simple proportion.

Figs. 1 (a) and (b) illustrate measuring apparatus of the type we have normally employed. Referring to Fig. 1 (a), the slightly inclined water-cooled disc has a pointed lip at the lowest part of its rim. Condensate accumulating on the underside of the disc runs down to the point and drips off. When time intervals between drops become equal, indicating that a steady state has been reached, a light aluminium cup is moved under the point to catch the drops over a measured period of time. Collection usually begins, and also ceases, just after a drop has fallen. The cup is moved away from the point while the pump cools down so that the cup can be removed for weighing.

The cup usually stands in accumulated oil on the flat base of the water-cooled test header and so may be assumed to have header temperature. This external oil is washed off before weighing.

The interval between drops may exceed half an hour and a complete test may take many hours and it is important that the heater input be kept constant for a considerable period before the actual collection of drops, as well as during collection. The result then indicates the steady backstreaming rate of the pump, any variation during warming up or cooling down being excluded by the method of measurement. Experience has shown that the results are reasonably repeatable.

Fig. 1 (b) illustrates a similar device employing a sector only of the complete disc for use on a large pump. It will be noted that the whole pump mouth is covered with a water-cooled disc from which a suitable sector has been removed. The collector plate is somewhat larger than the cut-away sector of the disc, to overlap it all round, but the area of the cut-away portion is of course the important quantity for calculation of total backstreaming.

Sometimes, for instance when the pump mouth is lightly baffled, the backstreaming rate may be much too low to be measured by the drop method. In such cases the authors have made some measurements by

holding a thin polished steel disc closely to the underside of the water-cooled collector plate by means of a magnet on top. The amount of condensate is too small to run on the disc face in such a case, and the disc is weighed to assess the condensate collected.

Other methods of backstreaming measurement which have been used[3] involve suspending a paper or foil collector above the pump mouth and weighing it before and after exposure to backstreaming. The authors have tested this method using the arrangement of Fig. 2 where the foil was hung lightly on one side of a shutter of an area similar to that of the foil so that a rotation of a spindle would expose either the collector or the shutter to the pump mouth. By this means the period of condensate collection could be limited to exclude pump warming-up and cooling-down periods. It has already been argued that only limited confidence can be placed in results obtained with a collector which is not temperature-controlled. Consider for example the loss by evaporation of 'collected' backstreaming from a collector at 25°C where the pump and test header cooling water is at 20°C. A typical pumping fluid might have a molecular weight of 400, and a vapour pressure of 10^{-6} mm. Hg at 20°C and 2×10^{-6} mm. Hg at 25°C which may be considered equivalent to a partial excess pressure of 10^{-6} mm. Hg. The net loss by evaporation may then be as high as

$$\mu = p\sqrt{\frac{M}{2\pi RT}} \qquad gm./sec./cm.^2$$

with the usual notation in c.g.s. units and assuming vaporisation and condensation coefficients of unity. On substitution:

$$\mu = 1.36\times981\times10^{-6}\sqrt{\frac{400}{2\pi\times8.31\times10^{7}\times298}}$$

For a collector area of 60 cm.^{2} this represents a total loss of

$$\mu\times60^2\times10^3 = 0.24 \qquad mgm./min.$$

If a loss approaching this calculated figure does in fact take place, then the results obtained by such a collector for a pump of 2 or 3 inch. bore may be considerably misleading as the total backstreaming

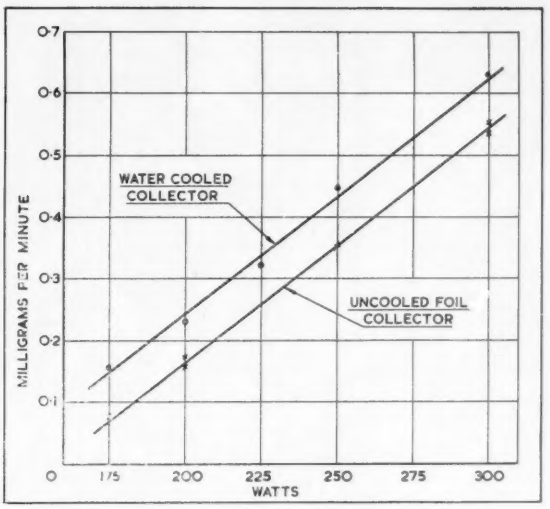
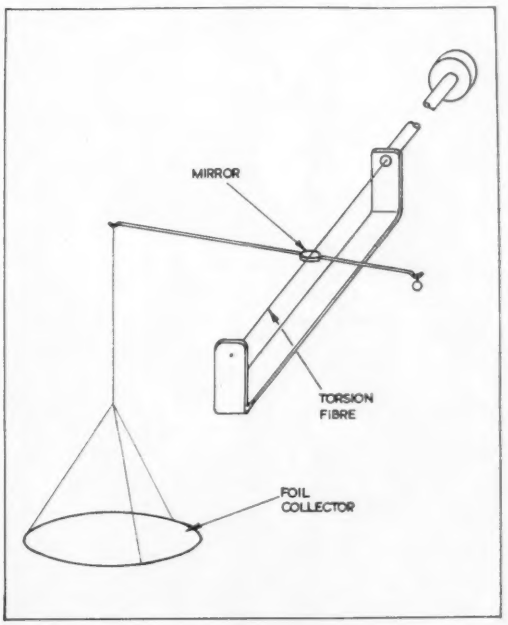


Fig. 3. Comparison of results given by cooled and uncooled collectors.

Fig. 4. Micro-balance and uncooled foil collector.



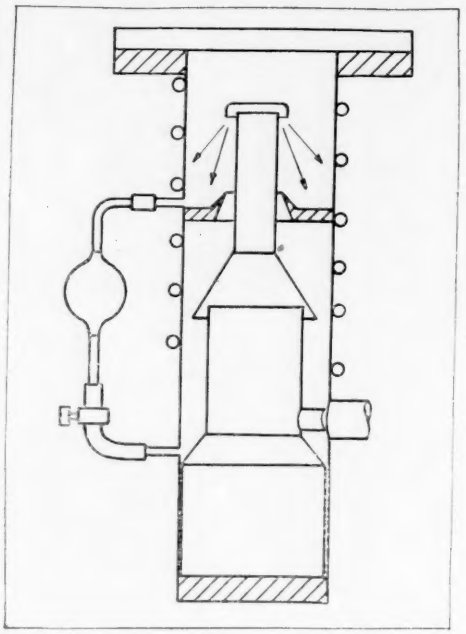


Fig. 6

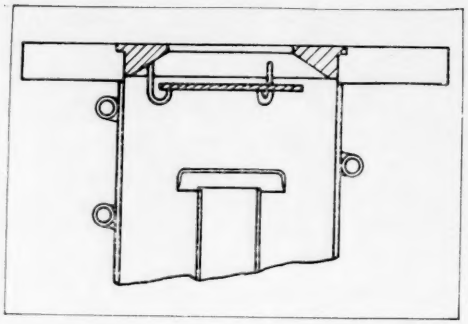


Fig. 7

Fig. 5. Backstreaming/heater input curve for Edwards 203 pump using Silicone 702 fluid.

Fig. 6. Measurement of vapour feed through top jet.

Fig. 7. Conduction cooled slip-in baffle for Edwards 203 pump.

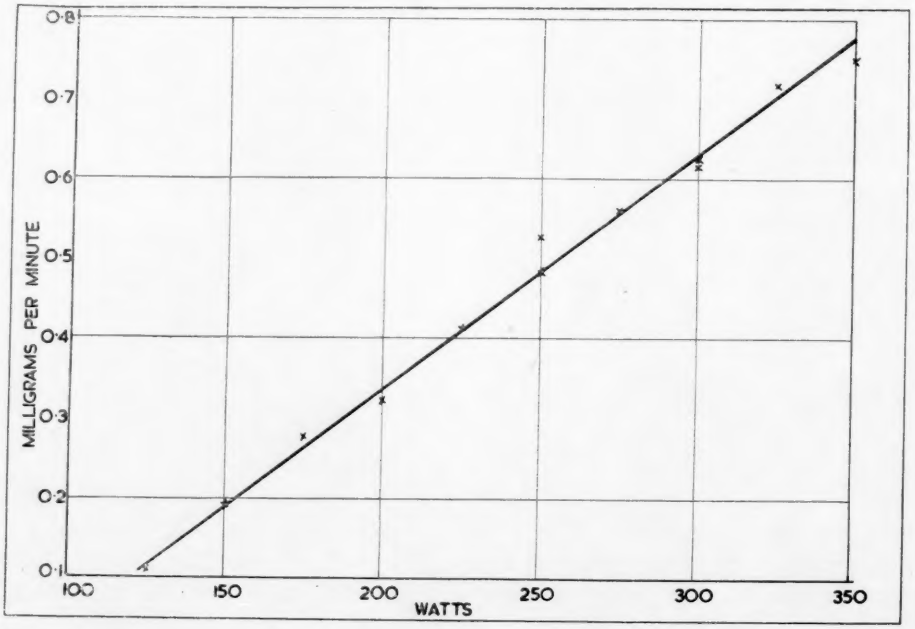


Fig. 5

from such a pump may well be only a fraction of a milligram each minute.

This is borne out by Fig. 3, in which are plotted the backstreaming rates for an experimental 2¼ inch. pump using Silicone 702 fluid at various inputs, as measured by a water-cooled collector and by an uncooled collector made of aluminium foil. It is clear that the results given by the foil collector are appreciably lower, and may be as little as 50% of the 'true' values at the smaller inputs. It is likely moreover that with a pump having a backstreaming rate intrinsically lower than that of the experimental pump the arithmetic difference in the results using the two methods would tend to remain the same and the percentage error greatly increase.

An uncooled collector made of paper was also tried using the same pump as before and gave results slightly lower even than the aluminium foil. In a further test with the foil removed altogether, the temperature of the supporting shutter was measured, at 250 watt input, and found to be 21.4°C when the pump-cooling water was at 17.2°C, so that the conditions were not unlike those assumed in the example calculated above. The absolute pressure—temperature characteristics of the fluid used are not sufficiently well known to permit calculation in the experimental case.

A type of collector used during early investigations is illustrated in Fig. 4. It consists of a light foil target hung from the arm of a calibrated torsion microbalance having a phosphor-bronze ribbon suspension and a mirror at the pivot to reflect a light spot onto a scale through a window in the test header. This type of collector is now considered to be unreliable for various reasons discussed above, but it has the considerable advantage that the instantaneous backstreaming rate at any moment is indicated by the rate of deflection of the balance so that variations in backstreaming rate throughout a test run can be studied. Some of these measurements will be referred to later.

The location of sources of backstreaming has been studied by other workers[3] by an interesting technique employing a diaphragm with a small central hole, supported above the pump jet assembly. Backstreaming molecules come through the hole in substantially straight lines and condense on a suitable collector above it in layers of varying thickness in a pattern which provides a virtual 'pin-hole photograph' of the sources. One form of collector used in this technique is a half-processed interference filter on which the pattern of oil layers can be 'fixed' after exposure by applying the remainder of the coatings. The variation of thickness of the oil layers can then be observed by the colour contrast they produce, and the locations and—it is also claimed—the relative intensities of the backstreaming sources are indicated. It seems that this last claim can only be supported if the various sources are assumed to have no directional character, an assumption which we do not believe to be completely justified.

Some work has also been reported in which a number of metal targets have been suspended across the mouth of the pump[4], and the oil condensed on each target measured separately. Although this method is liable to error due to uncertain temperature differences, as discussed above, it does serve to determine the distribution of backstreaming across the pump mouth and its variation under different conditions. The results show among other things that in the pump used for the experiments the backstreaming rate per unit area was about twice as high near the pump wall as on its axis, a conclusion which does not necessarily imply that the main source of backstreaming is near the cooled walls of the pump, since it could also correspond to the existence near the pump axis of a major source with an outward directional characteristic.

Results Obtained with Cooled Collector

Before discussing the sources of backstreaming and methods of reducing it, we shall present some results obtained with the measuring device using a collector at pump-wall temperature. These measurements were all made with a 2¼-inch. bore three-stage pump (Edwards Model 203) having the sealed top cap and baffle system above its boiler which are discussed later, but otherwise of conventional design and having no baffles above its top jet.

The curve in Fig. 5 shows backstreaming rate against heater input using Silicone D.C.702 fluid

(standard input is 250 watt). Possible mechanisms of backstreaming are discussed at length later and it is sufficient to point out here that as heater input and boiler pressure are increased both the mass flow and the vigour of the vapour issuing from the pump nozzles increase and the number of molecules suffering accidents leading to back migration would be expected to rise.

The pump used was separately tested with a gutter fitted below the top jet (see Fig. 6) so that the mass rate of vapour feed to the top jet could be measured by collecting the condensate. This quantity was found to be 4.0 gm./min. at standard heater input, corresponding to the use for this jet of about 25 watt out of the 250 watt total input. We can thus state that for this pump only about one molecule backstreams out of each ten thousand issuing from the jet. Results for various heater inputs are given in Table I.

Table 1. **Comparison of Backstreaming Rate with Top Jet Vapour Feed for Silicone 702**

Heat input watt	*Backstreaming Rate mgm./min.*	*Top Jet Feed mgm./min.*	*Proportion Backstreaming 1 part in*
150	0.187	2020	10800
175	0.262	2710	10300
200	0.335	3290	9800
225	0.410	3730	9100
250	0.484	4050	8400
275	0.558	4360	7800
300	0.632	4650	7400

A simple slip-in baffle cooled merely by contact with the pump walls is a standard accessory for the type of pump tested. When this was fitted (see Fig. 7) the residual backstreaming rate was very low and was measured by the method, already described, using a thin polished steel disc clamped magnetically to the underside of the collector. After running continuously for three days the visible deposit on the disc was only a very faint film in the centre and the backstreaming rate (with Silicone D.C. 703 at 250 watt) was found to be about 0.00014 mgm./min. The baffle had therefore reduced the backstreaming rate by more than 3,000 : 1.

The curve in Fig. 8 shows backstreaming rate against pressure in the pump mouth when a flow of air is admitted into the test header through a needle valve. It can be expected that some backstreaming molecules would be turned back by repeated collision with air molecules and in this particular case the effect became apparent at pump inlet pressures above about 10^{-4} mm. Hg. The effect would naturally be more marked if the collector were further away from the pumping stage giving more opportunity for intermediate collisions.

The two sets of curves in Fig. 9 show backstreaming rate against heater input using several different pumping fluids. The molecular weights of some of these fluids differ considerably so that a comparison of backstreaming rates in milligrams/minute gives a somewhat different picture from that given by a comparison of backstreaming rates measured in numbers of molecules a minute. The curves have accordingly been plotted both ways.

The points from which the curves were drawn have been omitted from Fig. 9 for clarity, but the spread of the results was similar to that shown for Silicone D.C. 702 in Fig. 5 and for each fluid the points were found to lie quite closely on a straight line over the range of inputs chosen.

At standard heater input (250 watt) there is only about a 3 : 1 difference between the worst and best fluids, even though the group contains fluids of such different vapour pressures that there is about a 30 : 1 difference in the ultimate pressures produced by the least and most phlegmatic of them in the pump used.

On the whole the fluids giving the lowest backstreaming rates are those known to give the lowest boiler pressures (and thus the least vigorous jets and the lowest limiting backing pressures) at any particular input. Thus when a pump of the same type was tested at 250 watt with Apiezon C and with Silicone D.C. 702 the limiting backing pressures were found to be 0.20 and 0.73 mm. Hg respectively. The heater input with Silicone 702 had to be reduced to 135 watt to reduce the limiting backing pressure to the value given with Apiezon C at 250 watt.

This suggests that for a comparison of backstreaming rates of two fluids on the basis of equal boiler pressures, the rate for Apiezon C at 250 watt should be compared with that for Silicone 702 at a much

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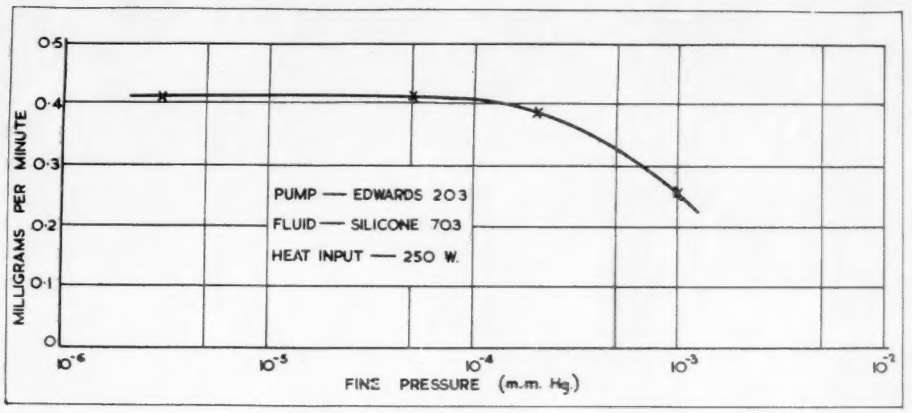

Fig. 8. Variation of backstreaming rate with fine pressure.

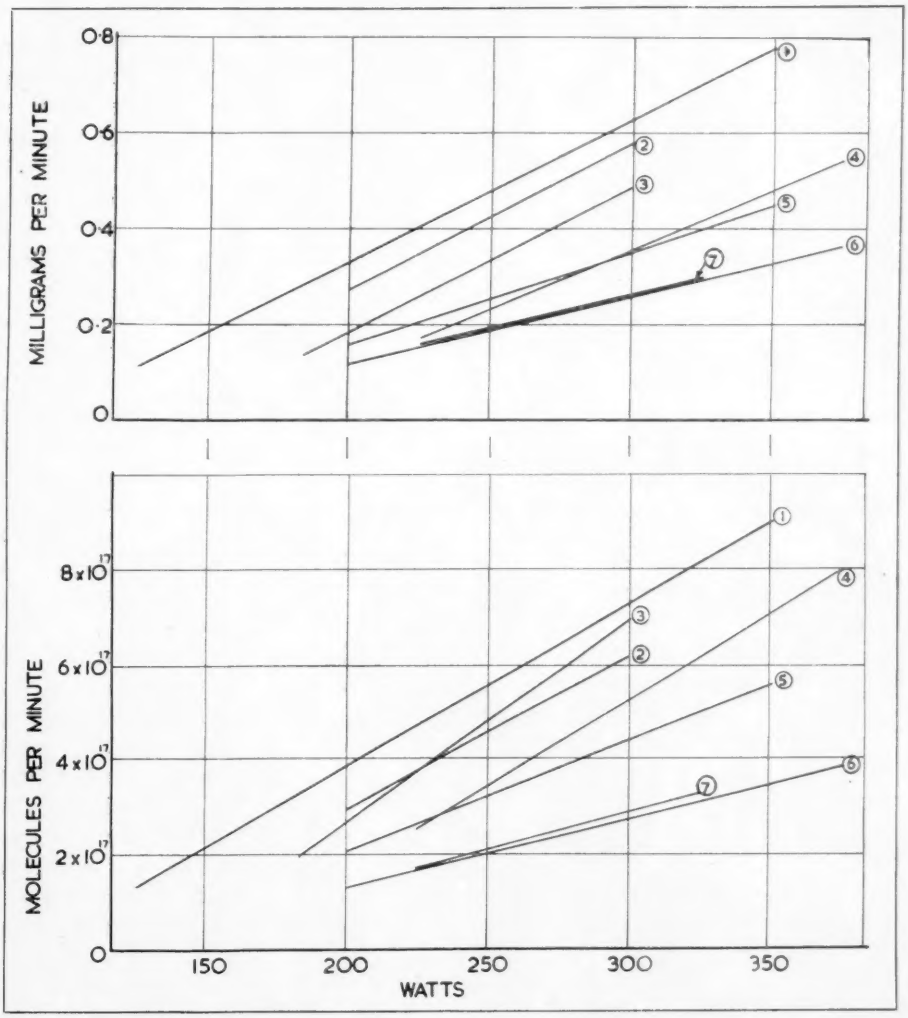

Fig. 9. Backstreaming/heater input curves for various fluids in Edwards 203 pump.

Curve	*Fluid*	*Molecular Weight*
1	Silicone 702	530
2	Silicone 703	570
3	Octoil S	426
4	Tri-Xylenyl Phosphate	414
5	Apiezon B	468
6	Apiezon C	574
7	Exptl Silicone	546

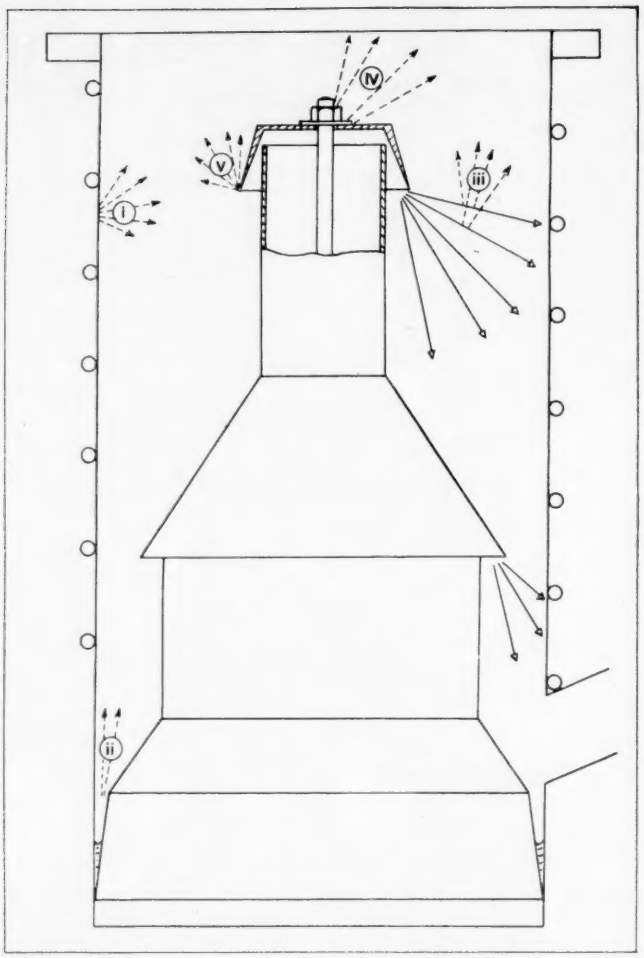

Fig. 10. Possible sources of backstreaming in a diffusion pump.

lower input. Reference to Fig. 9 shows that on this basis the rates for the two fluids become almost the same.

We may conclude therefore that, with the fluids generally used, the backstreaming rate depends as much on the boiler pressure produced at the selected heater input as on the chemical group to which the fluid belongs.

SOURCES OF BACKSTREAMING

Fig. 10 illustrates some of the possible sources of backstreaming which are known to exist, though their relative importance may vary greatly from one pump to another. Using the numbers in Fig. 10:

(i) Evaporation in excess of that appropriate to equilibrium at cold wall temperature may occur from the cooled pump wall, both because the considerable heat transfer through the low conductivity oil film to the wall must lead to a temperature higher than wall temperature on the surface of the oil film where the jet is impinging, and because of incomplete accommodation of the impinging vapour to this surface temperature.

(ii) We have encountered some pumps in which condensate, returning to the boiler and tending to boil just before entry to the boiler, has spat small drops of hot liquid right up through the vapour jets and out of the pump mouth. Though rather different from true backstreaming such spitting certainly gets measured along with the backstreaming vapour and would also produce similar ill effects in the system. Fortunately it can be readily avoided by good pump design once the danger is appreciated.

(iii) Inter-collision of vapour molecules, particularly in the top layer of the vapour jet after it has left the nozzle, may lead to the back migration of some of them. The colliding molecules may have re-evaporated from the cooled pump wall.

(iv) Even apparently insignificant leaks through the top cap, such as might be provided by the fixing nut and washer illustrated, can lead to severe backstreaming. It is likely that the pumping fluid seeps up the thread or across the washer as liquid and evaporates invisibly as it emerges into the high vacuum above the cap.

(v) The lip of the top stage jet cap is an important

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source. In some cases this lip is covered with visible liquid which must of course be very hot, and from which severe backstreaming arises. Even when the top cap lip is apparently dry however, this region remains an important source and will be discussed at greater length later.

The possible sources i, ii and iii cannot usefully be discussed further at present. Source ii should never be allowed to arise and deserves no more comment. Sources i and iii deserve further attention but they are known to be minor compared to sources iv and v which have been responsible for most of the backstreaming from commercial vapour pumps and which will be discussed more fully below.

Top Jet Cap and Jet Assembly Leaks

A method of fixing the top jet cap like that illustrated in Fig. 10 is very commonly used. It is not unusual, when such a pump is warming up, to observe liquid seeping out from under the retaining nut and evaporating as it spreads over the hot cap. In most cases, as full working temperature is approached, the liquid patch diminishes and finally disappears. However, even though the nut may appear to be tight and well fitting, a certain amount of liquid seepage may still be taking place, the liquid evaporating invisibly as it emerges into the high vacuum outside the cap (repeated expansions and contractions of the assembly usually tend to cause loosening which worsens the condition). It would seem most probable that any leakage from under the retaining nut occurs as liquid, particularly since, for reasons given in the next section, there is usually a film of working fluid condensed on the inside surface of the jet cap.

Repeated measurements of backstreaming rates on a pump of this construction gave completely random results, the worst rates being as much as ten times greater than the best. When the top cap was positively sealed however the fluctuations disappeared and repeated measurements gave low and consistent values for the backstreaming rate. All numerical results quoted in this article were obtained with sealed caps.

Seepage similar to that described for the top cap nut has been observed further down the jet assembly where constructions making it possible have been used. While we have no evidence that moderate seepage below the top vapour jet contributes significantly to backstreaming we believe these constructions are unnecessarily dangerous and should be avoided.

Wet Running

As already mentioned, in the worst cases a visible rim of liquid may be seen around the nozzle lip, liquid drops may occasionally be blown off, and there may also be runnels of liquid on the outside of the vapour tube below the jet cap. Liquid on the hot cap visible through the pump mouth is a vigorous backstreaming source.

Liquid under the top cap may arise through direct splashing from the boiler. It may also arise due to condensation inside the cap, since the latent heats of the common fluids are low and radiation heat loss from the cap is continuous. In some cases where vapour flows are vigorous some of the liquid condensed due to heat loss on the inner walls of the

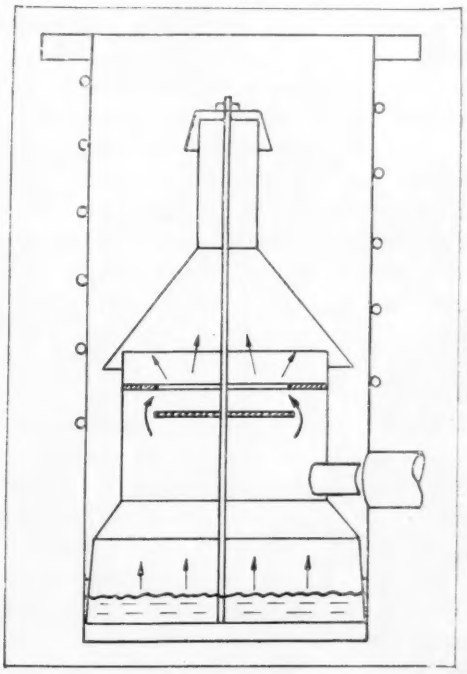


Fig. 11. Splash-baffle inside jet assembly of diffusion pump.

vapour tube below the jet cap may be blown by the vapour stream up the vapour tube and out onto its outer surface.

A very powerful method of promoting dry running of the top stage, and one we have used extensively*, is to provide a simple baffle formation inside the main vapour chimney some distance above the pumping liquid surface. Fig. 11 shows a typical arrangement. The first and most obvious purpose for these 'splash-baffles' is to intercept and precipitate spray coming from the boiler and this they do most effectively. A second purpose is to subject the vapour on its way to the various nozzles to some degree of turbulent throttling. The thermodynamic properties of the common pumping fluids are not well known, but we believe them to be such that even an isentropic expansion tends to have a 'drying' effect (the opposite to the effect with steam) so a turbulent expansion should have a significant drying action.

The provision of splash-baffles has time and again improved a visibly wet running top stage so that no wetness could be seen and the drying effect on the lower stages has also been most pronounced. The influence the combined effects of splash-baffles can have on the backstreaming rate is illustrated by measurements made with a 2¼-inch. bore three-stage pump (Edwards Model 203) using Silicone D.C.703 fluid. The rate was 0.43 mgm./min. with a splash-baffle fitted and 1.14 mgm./min. without a baffle.

Any means of reducing the heat loss from the jet assembly naturally helps to keep the jets running dry and an obvious step is to provide a bright exterior surface of low emissivity. To demonstrate how important this can be, some tests were made with top jet caps of different emissivities. The results of these tests are presented in Table II and effectively demonstrate the desirability of a surface of low emissivity.

The increased backstreaming consequent upon increased radiation heat loss is by no means necessarily accompanied by visible wet running and this suggests that, over a certain range of operating conditions, a very thin liquid film may be flowing down inside the cap and flashing off invisibly in a random vapour cloud as it emerges onto the lip.

* Brit. Patent No. 700978.

Other methods which have been used in an attempt to ensure completely dry running include the provision of radiation shields around the top jet cap and lower parts of the jet assembly, and the heating of the top jet cap either by conduction (*e.g.* a central copper rod from the boiler base, or the use of a thick walled cap of highly conducting material to carry heat from the crown to the lip[3]) or by electrical heating.[4]

Even when dry running would seem to have been assured by such means, however, the backstreaming from the region of the lip of the top jet cap, though greatly reduced in amount, still contributes the major part of the total residual backstreaming, assuming of course that the jet cap and interior are leak free.

To confirm this statement an experimental pump of 9-inch. bore with a fairly high backstreaming rate was provided with an electric heater on its top jet cap,

Table II. **Effect of Top Jet Cap Material on Backstreaming Rate**

Pump	*Jet Cap Material*	*Estimated Emissivity %*	*Backstreaming Rate mgm./min.*	*Fluid*
203 (2¼ in)	Aluminium	10	0.48	Silicone D.C.702
	Mild Steel	50	0.78	
	Nickel-Plated Copper	10	0.32	Experimental Silicone Fluid
	do. Graphited	100	0.57	
903B (9 in)	Nickel-Plated Copper	10	17.0	Silicone D.C.702
	do. Graphited	100	26.6	

and a curve of backstreaming rate against heater input to the jet cap was obtained. Initially the backstreaming rate fell sharply as the heater input was increased but after a certain value of input had been reached, at which completely dry running was presumably obtained, further increases in heater input had no further effect on backstreaming rate. It was confirmed that the bulk of the considerable residual backstreaming originated from the jet cap lip by applying a device, to be described later, to trap backstreaming from the lip at source. A very great reduction in

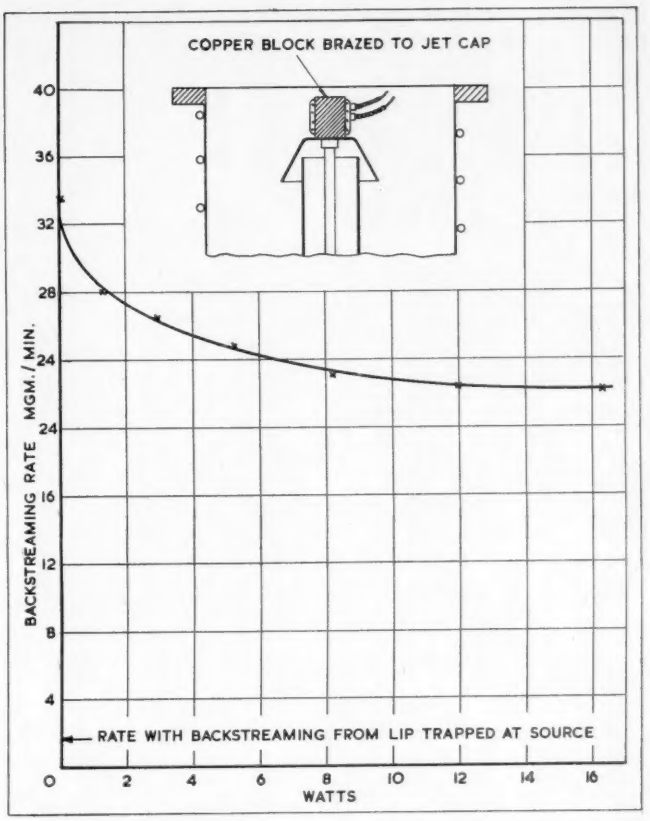


Fig. 12. Effect of heating top jet cap of 9-inch pump.

backstreaming rate was than recorded and similar reductions have been obtained in a large number of apparently dry running pumps by the application of the same device. The results obtained with the heated jet cap pump are shown in Fig. 12 which also shows the arrangement of the heater as an inset.

Residual Backstreaming from the Nozzle Lip

An attempt will now be made to suggest a reason why randomly moving vapour appears at the lip even of a completely dry nozzle.

It is well known that nozzles designed to accelerate air to high speeds at very low pressures, for instance for low pressure wind tunnel work, present many special problems. One of these is provided by the excessively thick boundary layer which forms along the nozzle wall under such conditions.

The boundary layer is a region adjacent to the nozzle wall where velocity gradients are very high and where the motions of the individual molecules are influenced and disturbed by collisions with the wall and the other molecules already moving randomly. There is then in every high velocity nozzle a central region or core where the molecules have been accelerated to reasonably uniform velocities in the desired direction, and a boundary region adjacent to the wall containing molecules of widely assorted velocities and increasingly random motion as the wall is approached.

Boundary layer thickness naturally tends to increase as the nozzle lip is approached both because the effect of the undesirable molecule–wall and molecule–molecule collisions is cumulative, transferring more and more molecules to the boundary layer as the expansion through the nozzle proceeds, and because molecules travel on the average greater distances without collision at the lower pressures near the nozzle exit and so a randomly travelling molecule from the boundary layer has more chance of penetrating into the main vapour stream. At ordinary pressures, however, boundary layers are usually very thin.

At very low pressures, where molecular mean free paths are much longer, boundary layer growth in a nozzle is much more rapid and it is found, when using a nozzle to accelerate the air stream for a very low pressure wind tunnel, that only a small central core of air is moving with the desired uniform high velocity at the nozzle mouth and that most of the nozzle mouth area is occupied by the boundary layer. In such cases boundary layer growth can be controlled by sucking away the layer through slots in the surface against which it is forming, and this technique has been effectively applied.

We believe that similar boundary layer growth is to be expected in the nozzles of vacuum vapour pumps

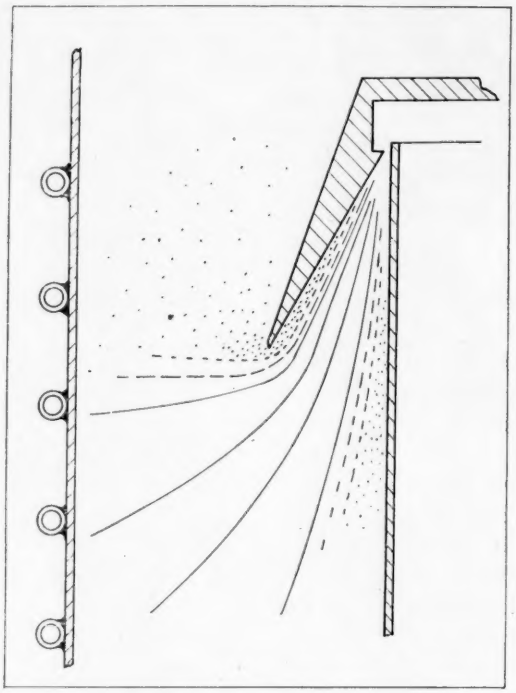
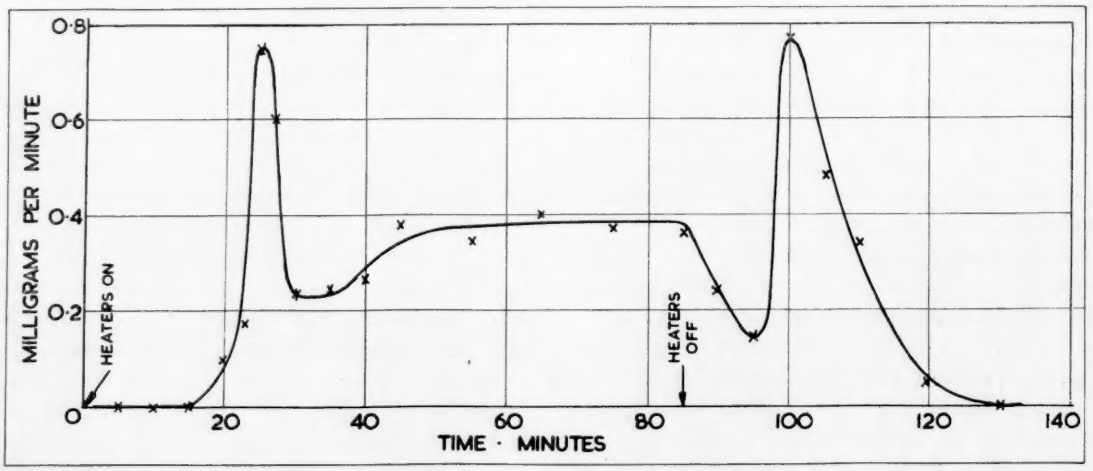


Fig. 13. Representation of boundary layer formation in diffusion pump nozzle.

Fig. 14. Backstreaming peaks during warming-up and cooling-down periods, using micro-balance and uncooled foil collector.



and that the conditions must be something like those illustrated diagrammatically in Fig. 13. Here the vapour jet is shown as consisting of a central region where the molecules are moving substantially with the desired velocity and direction, and two regions near the nozzle walls where velocity gradients are severe and there is a great deal of random molecular motion.

In this case there must issue from between the nozzle lip and the well-formed part of the vapour jet a random cloud of molecules of such mixed velocities that multiple collisions resulting in migration in all directions are quite inevitable, so that the lip of even a dry running nozzle is an important backstreaming source. Methods of controlling this source will be discussed below.

Warming and Cooling Periods of Pumps

To conclude the survey of backstreaming sources it is worthwhile mentioning the special conditions when the vapour pump is just coming into operation while the boiler is being heated up and when the pump is just ceasing to operate after switching off the heaters.

During warming-up a confused cloud of vapour driven by an inadequate boiler pressure emerges from the pump nozzle into a region of comparatively high air pressure. This vapour is losing much heat to the jet assembly which must be warmed to operating temperature largely by the vapour stream, so the jet and vapour tube run very wet. A severe backstreaming peak is to be expected.

During cooling-down the vapour jet must, before it ceases entirely, pass through a stage when it is so feeble that air penetrates back from the fore side and conditions exist similar to those on warming up, so that a second backstreaming peak can be predicted.

The micro-balance method of measuring backstreaming described earlier, though unreliable for absolute measurements, is able to reveal comparatively any changes in backstreaming rate during a particular run. The curve shown in Fig. 14 was taken by this method using a 9-inch. diameter diffusion pump and illustrates the peaks which are to be expected. These peaks are severe but very transitory and their importance should not be over emphasised.

EFFECTS OF VAPOUR JET DIVERGENCE ON PUMPING SPEEDS

The coherent part of the vapour jet shown in Fig. 13 has been illustrated as diverging sharply as it leaves the nozzle lip. This divergence is characteristic of vapour jets emerging into regions of very low pressure and it is particularly severe with the vapours generally employed in oil diffusion and booster pumps where the ratio of specific heats of the vapour is nearly unity, so that the upper layer of the high-velocity vapour stream appears to be very nearly horizontal.*

In the case of the powerfully fed nozzles of vapour booster pumps the divergence can of course be more severe than with diffusion pumps and the vapour in the top layer of the jet often travels at a slight angle above the horizontal, so that the vapour reaches the condensing wall with an upward component of momentum. Condensing walls of conical form (see Fig. 15) are frequently used in booster pumps and in such cases the component of momentum along the wall, against the pumping direction, delivered by vapour molecules in the top layer is even more pronounced.

The vapour jets of booster pumps are so dense that it is quite likely for a proportion of the air diffusing into them to be swept to the wall before it has penetrated below the region where the reversed pumping action exists, and this air will obviously re-emerge at the top of the vapour jet instead of being pumped. Some results obtained during tests of diffusion pumps of conical and parallel form suggest that the speeds even of these pumps can be influenced by the angle at which the top layer of the vapour jet strikes the condensing wall; this implies that the deliberate contouring of the wall to ensure a favourable angle of impingement of the whole jet might be advantageous in some cases. During development work on vapour booster pumps working in conical bodies, cases have been encountered where the speeds at low pressures have

* A very extensive survey of the characteristics of jets in vapour vacuum pumps has recently been made, Ref. 5.

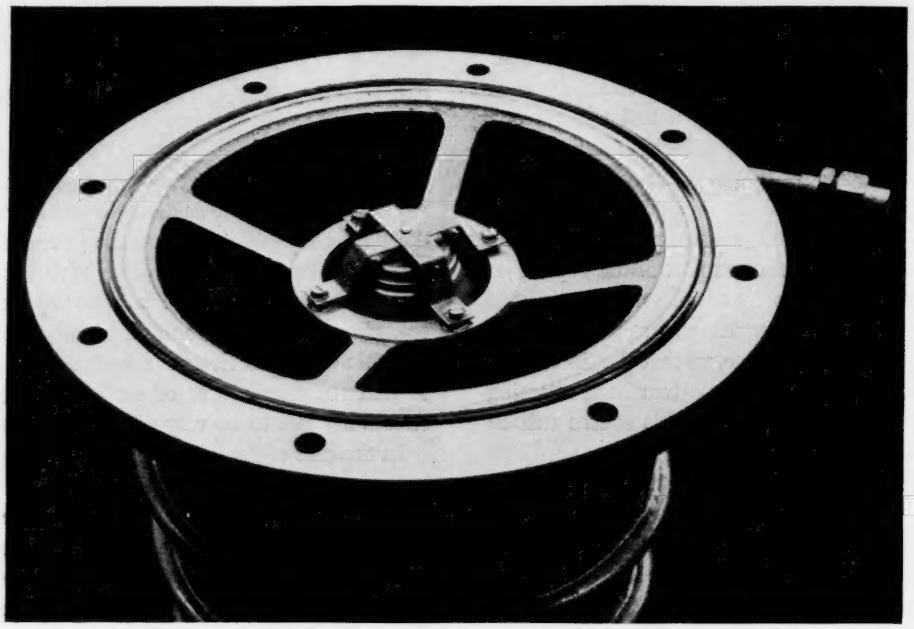
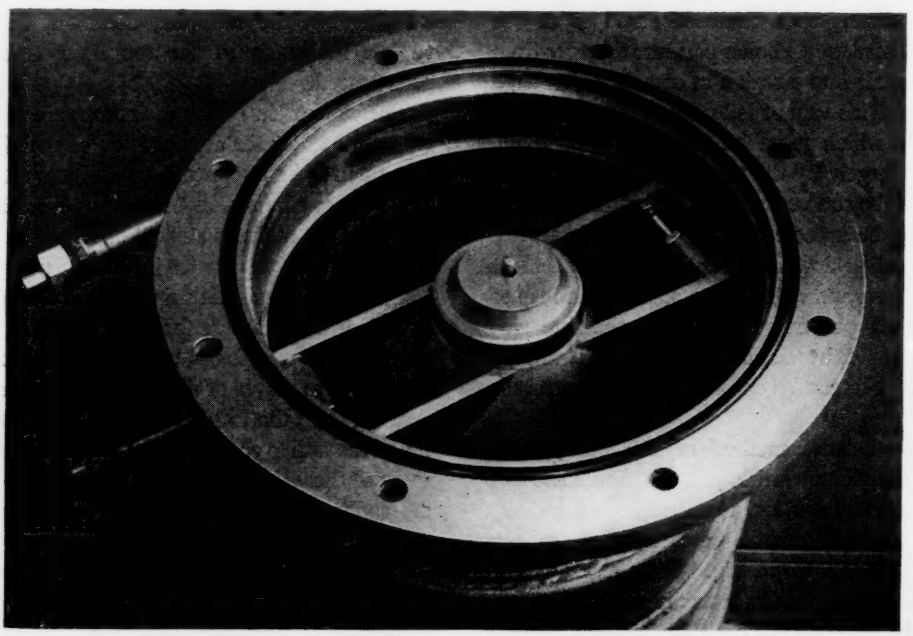


Fig. 16*b* (i) Guard ring as fitted to 18B3 booster pump (above).
(ii) Guard ring as fitted to 9-inch. diffusion pump (below).



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been depressed to the order of only 50% of the anticipated values, the anticipated results being fully achieved at higher pressures where the vapour jet would be expected to cease to diverge so severely.

The effects just discussed are only indirectly related to backstreaming as defined earlier (though very severe backstreaming can also influence pump speeds and the two effects cannot always be clearly separated), but they have been described because they led the authors to consider whether the troublesome top layer of the vapour jet could be skimmed off after it had left the nozzle, and the method adopted for this purpose proved also to be a very effective method of backstreaming control.

CONTROL OF BACKSTREAMING

Cooled 'Guard Ring' Control Device

To control the effects described in the last section it was decided to attempt to 'skim off' the top layer of the over-divergent vapour jet after it had left the nozzle lip. To do this a cooled ring of copper was provided around and just outside the nozzle lip and extending downwards for a short distance below the lip. It was hoped that if the undesirable top layer were condensed away, there would not be a too severe second divergence as the rest of the jet passed under the ring. Fig. 16 (a) attempts to illustrate this expectation.

Guard rings* effectively improved the speeds of the unsatisfactory vapour booster pumps at low pressures. Before their use, speed measurements in the 1–10 micron Hg pressure range on a number of prototype pumps gave not very repeatable results, varying around an average of about 750 litres/sec. Since their adoption, pumps of this type (Edwards Model 18B3) have yielded speeds quite closely grouped around about 1,000 litres/sec. in this pressure range.

The rings were also surprisingly effective at reducing the backstreaming rate in both booster pumps and diffusion pumps so that this quality alone often justified their use where the anticipated pumping speed was already being obtained without them. We

* Brit. Patent Appl. No. 25315/54

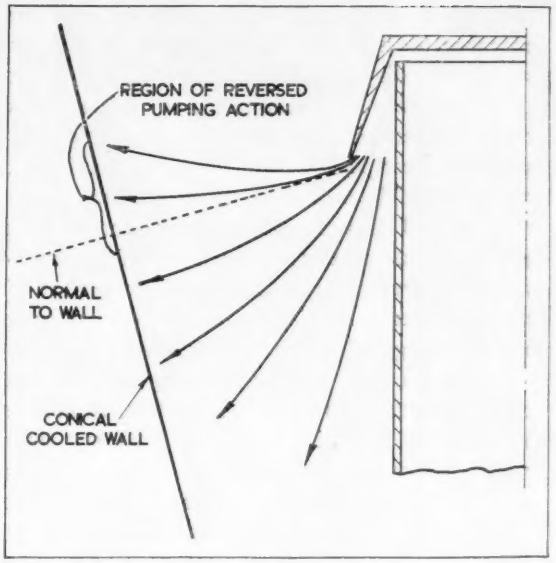
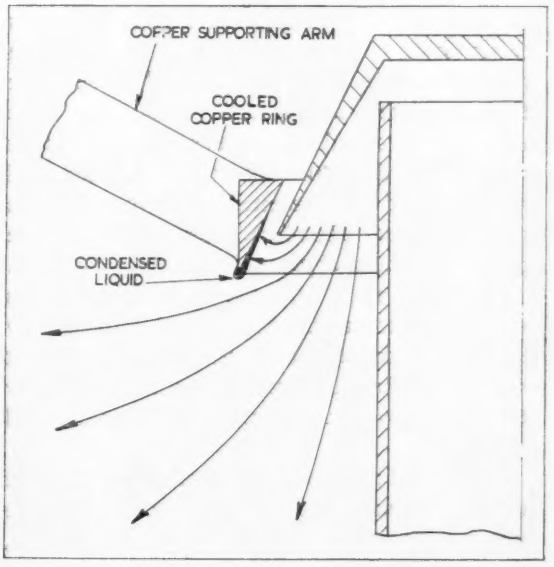


Fig. 15. Over-divergence of jet in vapour pump with conical wall.

Fig. 16*a*. Cooled guard ring to intercept over-divergent layer of vapour jet.



have described ways in which random vapour can arise from the nozzle lip and it is obvious that a cooled surface extending downwards to reach the well formed part of the vapour jet must intercept the bulk of the random molecules in the region. It was surmised that some might escape, however, up between the ring and the outside of the nozzle, so on later versions the ring was extended upwards until it became a complete cap over the nozzle and this resulted in a small but worthwhile improvement in backstreaming rate. Fig. 16 (b) shows an open guard ring for an 18B3 booster pump and a closed guard ring for a 9–inch. diffusion pump.

The application of the guard ring has, in some cases, reduced the backstreaming rate to as little as one-thirtieth of its initial value. The very great reductions obtained provide strong evidence that the region of the nozzle lip is the paramount source of backstreaming even when the nozzle runs visibly dry, assuming cap leaks and other gross sources have been eliminated, and justify the attention given to this region earlier in the paper. The effect of the guard ring on backstreaming rate is shown for a number of pumps in Table III.

With regard to pumping speed, it is only the very heavily fed jets of booster pumps, where admittance coefficients at low pressure are abnormally low, that benefit strikingly from the application of the guard ring. With diffusion pumps there is usually much less margin for improvement and although the admittance coefficient may be somewhat increased the structure of the guard ring and its cooling members to some extent reduces the admittance area and obstructs the pump mouth; this is particularly the case with small pumps where a high percentage of the pumping area may be obscured. The effects of the guard ring on the speeds of a number of pumps are shown in Table IV.

Early guard rings were water-cooled, and it was possible to measure the rate of heat extraction by temperature rise and water flow measurements, but it was later found more convenient in practice to cool by conduction through copper struts to the water-cooled pump wall.

If the guard ring intercepts an unnecessarily large amount of the vapour stream, it will receive heat at an unnecessarily high rate. If the ring becomes too hot, vapour will randomly evaporate from the condensate on its surface and it will itself become a source of backstreaming. To permit the selection of the slimmest possible copper cooling struts it is helpful to know how hot the ring may be safely allowed to run and how much it should overlap the nozzle lip for efficient backstreaming control.

A curve of guard ring temperature against backstreaming rate for a 9–inch. pump using Silicone D.C. 702 fluid is shown in Fig. 17. Silicone 702 is one of the less phlegmatic of the high vacuum pumping fluids so that a guard ring having a low enough temperature with this fluid would be amply safe with the more phlegmatic fluids such as Silicone 703 or Apiezon C. It is seen that the temperature should not exceed about 35°C and as the vapour pressure of this fluid (ionisation gauge air equivalent measured above the pump) is about 8×10^{-5} mm. Hg at 35°C and about 1.5×10^{-4} mm. Hg at 40°C the result is about what would have been expected.

A typical guard ring on a diffusion pump runs at about 30°C and is continually dripping with condensed oil. The droplets are usually blown off onto the pump wall by the vapour stream and seem to cause no trouble.

The effect of varying the vertical overlap between the jet cap and the guard ring has not yet been fully investigated. However, in one series of tests on a 9-inch. bore pump the overlap was set successively at about $\frac{1}{8}$, $\frac{1}{4}$ and $\frac{3}{8}$ inch and each change reduced the backstreaming rate, the last rate being less than half the first. The guard ring must extract more heat and become hotter as the overlap is increased, and may also rob the jet of too much useful vapour, but the limits have not yet been explored. To quote examples of the heat extraction rate found satisfactory in practice, the ring round the top jet of a 9–inch. diameter three-stage diffusion pump with a 2.25 kW heater input extracts about 15 watt, and the ring used on a three-stage booster pump with a 9–inch. mouth and a 6.4 kW heater input extracts about 80 watt.

In the case of the booster pump impractically large cooling struts would be required to maintain the guard ring temperature ideally low and a higher running temperature has therefore been accepted. Even so

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the speed improvement reported earlier has been obtained and a large reduction in backstreaming rate achieved (see Table III).

Use of Baffles with Guard Ring

The guard ring greatly reduces backstreaming, but does not entirely eliminate it, and a conventional baffle remains necessary to give complete safety from condensation of pumping fluid in the pumped system. The load on the baffle is however, very greatly reduced and the protection it affords is correspondingly greater. We find much less condensation on baffles and baffle valves, the visible wetting being far less extensive on the surfaces of the baffle structure. Most baffles depend on vapour molecules travelling straight without direction changes due to inter-molecular collisions, and are naturally more effective if the concentration of vapour molecules beneath them is greatly reduced.

Control by Temperature Gradient along Nozzle Walls

Having concluded that the backstreaming arising at the nozzle lip may be due to the emergence onto the lip of a film of hot liquid and also to the emergence from under the lip of the vapour jet boundary layer of random molecules, we decided to investigate the possibility of dispensing with the guard ring by preventing the emergence of hot liquid onto the nozzle lip and by reducing or preventing the formation of the boundary layer within the nozzle.

Low pressure nozzles for handling permanent gases have in some cases been provided with slots so that the boundary layer could be pumped away as it formed, and considerable control has apparently been obtained by such means.

Table III. **Effect of Guard Rings on Backstreaming Rates of Various Pumps**

Pump Diam. Inches	*Input Watt*	*Backstreaming Rate—mgm./min.*		*Reduction*
		Without Guard Ring	*With Guard Ring*	
16 (a)	5200	70	5.5	12.7 : 1
9 (a)	2250	11.3	0.55	20.6 : 1
6 (a)	1300	1.5	0.15	10 : 1
9 (b)	2250	17	0.6	28.4 : 1
2¼ (b)	250	0.48	0.025	19.2 : 1
9 (c)	6400	170	10	17 : 1

(a) Fractionating diffusion pump using Apiezon C
(b) Non-Fractionating diffusion pump using Silicone 702
(c) Booster diffusion pump using Aroclor 1254

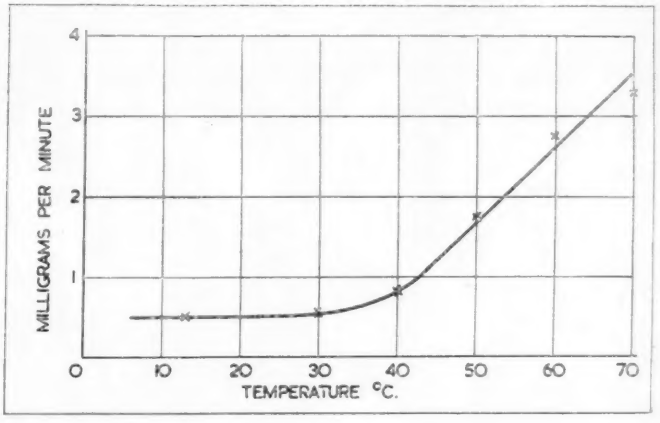


Fig. 17. Effect of guard ring temperature on backstreaming rate.

Table IV. **Effect of Guard Rings on Speeds of Various Pumps**

Pump Diam. inches	*Pumping Speed for Air—lit./sec.*		*Increase %*
	Without Guard Ring	*With Guard Ring*	
16	5200	5650	+8½
12	2800	3100	+11
9	1550	1510	— 3
6	650	550	—15
2¼	75	55	—27

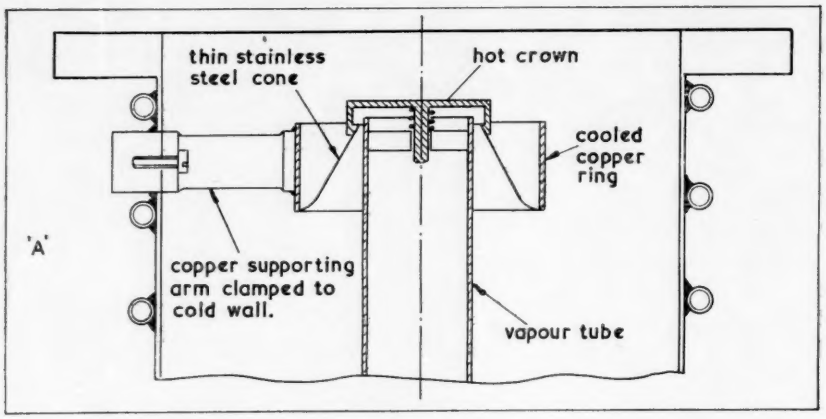
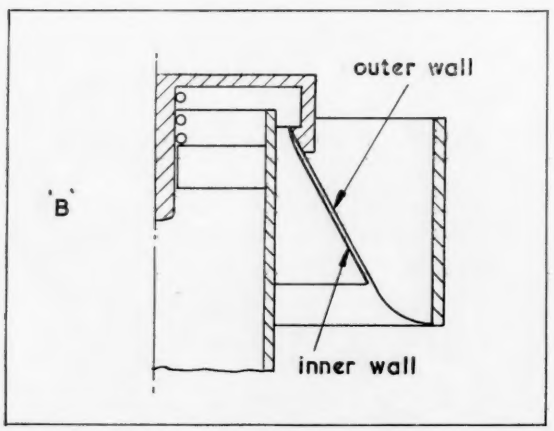


Fig. 18*a*. Experimental nozzle cap with cooled lip for 6-inch. pump.

Such a device would be over-complicated for use in a vapour vacuum pump, but the pump fluid vapours are ideally suitable for being 'pumped' by *condensation* and an attempt was therefore made to remove any boundary layer, as it formed, by controlled condensation on the inside of the nozzle wall. To attempt this method of control is to accept the 'wet running' of the nozzle—the emergence of visible liquid onto the nozzle lip—which we have been so careful to avoid in the past. But if the nozzle lip is sufficiently cool the emerging liquid will have too low a vapour pressure to cause any trouble. It is the emergence of *hot* liquid which must be prevented.

A number of nozzle constructions* has been devised all of which provide for the deliberate and controlled extraction of heat from the nozzle wall and thus completely reverse traditional design concepts (which all aim at keeping the nozzle as hot as possible). One of the simpler types of the new nozzle which will be described is generally like the illustrations in Figs. 18 and 19. The top or 'crown' of the nozzle cap is turned from any convenient material and may be of substantial section. The skirt is made of a thin material of fairly low thermal conductivity and the bottom extremity or lip of the nozzle is well cooled by any convenient means. We have used mild steel for the crown and stainless steel, only a few thousandths of an inch thick, for the skirt. These have been fixed together by brazing, and a substantial copper ring has been brazed to the extreme bottom lip of the stainless steel skirt to conduct away the heat, the ring itself being cooled by one or more copper struts having their outer ends clamped against the cooled pump wall and serving to support the nozzle rigidly and centrally in the pump.

* Brit. Patent Appl. No. 26899/56



In operation, the crown of the nozzle reaches the usual temperature of about 160–200°C depending on the characteristics of the pump and fluid used but the nozzle lip runs cool, about 35–40°C in pumps employed in the investigation, and there is a temperature gradient along the stainless steel skirt between these two extremes.

The rate of heat loss from the vapour stream to the nozzle wall is negligibly small near the nozzle crown but increases progressively as the cooled lip is approached and vapour from the boundary of the vapour stream must be condensing along a considerable

Fig. 18*b*. Experimental nozzle of the design shown in Fig. 18a, but using a double wall construction.

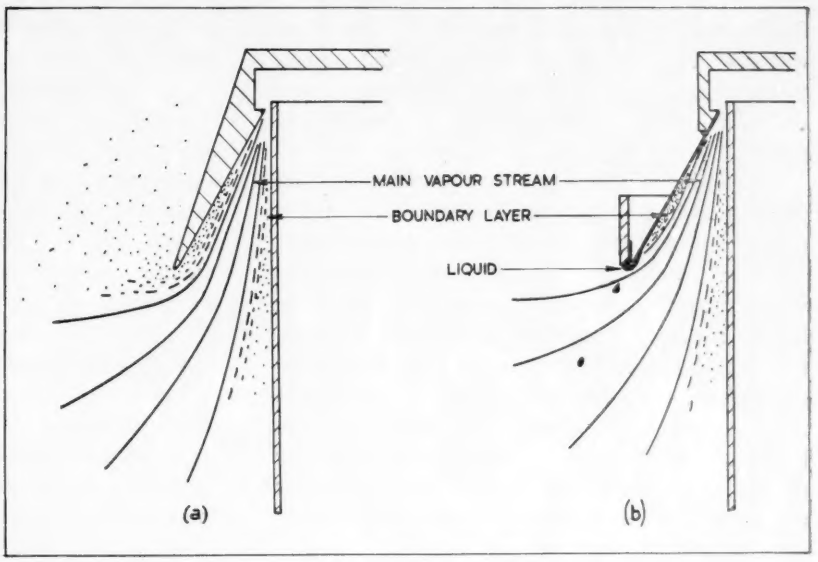


Fig. 20. Representation of suggested formation of vapour jet in (*a*) conventional nozzle; (*b*) nozzle with cooled lip.

length of nozzle wall above the cooled lip.

Ideally the boundary layer suspected to be present would be just completely condensed and no vapour would be extracted from the useful part of the vapour stream. In practice there is almost certainly some over-condensation near the lip and possibly some under-condensation higher up the nozzle. Fig. 20 contrasts a possible operating state in a conventional nozzle with a possible operating state in a nozzle of the type under discussion. These diagrams merely illustrate our conceptions; no attempt to check their validity experimentally has been made yet, but it is hoped to do so before long.

Experiments have also been made with cooled-lip nozzles similar to that shown in Fig. 18 (a), but having a second thin cone partially lining the outer conical skirt to limit the amount of vapour condensing on the skirt, as in Fig. 18 (b). This double-wall construction has proved useful as a means of controlling the temperature gradient without introducing the practical difficulties arising from the use of a single wall of very thin material.

From a practical point of view nozzles cooled progressively towards the lip appear likely to achieve the results intended even though they involve a complete reversal of conventional good practice — the careful avoidance of heat loss and of any wet running. They have so far been applied experimentally to 9–inch. and 6–inch. bore fractionating diffusion pumps. Table V contrasts backstreaming rates and pumping speeds so

Table V. **Effect of Fitting Guard Ring and Cooled-Lip Nozzle into 6-inch Diffusion Pump, Using Apiezon C Fluid at 1,300 Watt.**

Type of Top Stage	*Pumping Speed for Air lit./sec.*	*Backstreaming Rate mgm./min.*
Conventional Nozzle	650	1.5
do. with Guard Ring	550	0.15
Cooled-Lip Nozzle	600	0.10

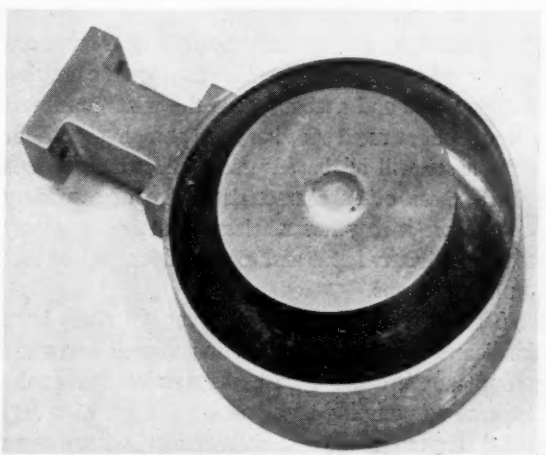


Fig. 19 Experimental nozzle cap with cooled lip for 6-inch. pump.

far obtained for the same 6–inch. pump when fitted with a conventional top jet cap alone, a jet cap and guard ring, and a condensing jet cap. Fig. 19 is a photograph of a condensing jet cap used in a 6–inch. pump.

Condensing jet caps may be clamped solidly to pump walls in a way not possible with conventional caps which must lose the minimum amount of heat and Fig. 18 illustrates a convenient construction which uses the firmly fixed jet cap to retain the whole interior assembly, a spring being provided to permit the necessary thermal expansion of the latter. Work on condensing jet caps is still in the initial stages and it is not yet certain how useful they will prove to be, but there would seem to be considerable scope for development and it is hoped to be able to give a fuller account of their operation and possibilities at a later date.

BACKSTREAMING AND OTHER PERFORMANCE CHARACTERISTICS

The methods that have been proposed for controlling backstreaming from the various sources have all been intended to operate *without seriously impairing other pump performance characteristics*. Before concluding the present discussion it should be pointed out that very low backstreaming rates can usually be readily achieved at the expense of other valuable characteristics by quite simple changes in pump design or operating conditions.

For example, a severe reduction of heater input can greatly reduce the backstreaming rate, but the limiting fore pressure and probably also the maximum mass throughput at rated speed and the pumping speed for light gases will all be adversely affected. In cases where these other characteristics are of secondary importance it may nevertheless be desirable greatly to reduce heater input and to provide improved fore pumping means.

Again, the use of a top jet cap comparatively large in diameter and with its lip situated well below the pump mouth can reduce backstreaming rate at the expense of pumping speed whilst the use of a very lightly fed top jet cap can achieve a low backstreaming rate at the expense of maximum mass throughput at rated speed and pumping speed for light gases.

Although growing understanding of the technique of pump design is continually increasing the extent to which all aspects of the performance can be kept simultaneously at a high level, the pump geometry and standard heater input adopted by a manufacturer do still represent a compromise intended to provide the best balance of performance characteristics for a wide variety of users.

CONCLUSIONS

The most common causes of excessive backstreaming in the past have been leaks in the jet assembly and particularly in the top jet cap, 'wet running' of the top stage, and the emergence of randomly travelling vapour from under the lip of the top jet cap even when this seemed to be running dry.

The importance of these causes has been demonstrated by measuring the change in the backstreaming rate when they are removed or controlled. Thus the sealing of a top jet cap in one case reduced the backstreaming rate by 10 : 1 and the promotion of dry running by the use of 'splash-baffles' above the boiler produced a further improvement of 3 : 1. Even pumps fitted with splash-baffles and exhibiting no visible wetness could be still more improved by applying heat to the top cap or reducing the emissivity of its surface. Finally the emergence of residual backstreaming vapour from the lip of even a leak tight and visibly dry nozzle has been confirmed by condensing this vapour on a 'guard ring' external to the nozzle and thereby obtaining a reduction sometimes as great as 30 : 1 in the backstreaming rate.

The authors have suggested that some of this residual backstreaming vapour may arise from thin condensed liquid films inside the nozzle and some may be produced from the 'boundary layer' of the vapour jet, and have described a nozzle intended to condense the boundary layer progressively and to cool the condensate as the lip is approached.

Typical examples of the influence on backstreaming rate of heater input, of fine pressure and of choice of pumping fluid have been presented and the experi-

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mental points on the various curves indicate a satisfactory degree of reliability and reproducibility for the measuring method used.

Various backstreaming measuring methods have been discussed and it has been argued and experimentally demonstrated that a lower backstreaming rate can be indicated by devices where the collector is not temperature controlled than by the method we have adopted, where collector, test header and the upper part of the pump body are kept at the same temperature. It is therefore important that, until a standard measuring method has become established, the measuring method is indicated whenever a backstreaming rate is quoted.

We wish to record our gratitude to Mr. A. S. D. Barrett, Technical Director of Edwards High Vacuum Ltd., for granting permission to publish this article.

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THE KNIFE-EDGE VACUUM SEAL

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Summary

A RELIABLE HIGH-VACUUM SEALING technique of wide applicability and capable of containing an ultra-high vacuum is described. This seal can readily be assembled or disassembled. It can withstand repeated and prolonged bake-outs as well as liquid nitrogen temperatures. Seals ranging from less than one inch to more than twelve inches in diameter have been constructed. A wide variety of materials have been joined.

This paper is based on a lecture contributed by the authors to the Second Symposium held by the Committee on Vacuum Techniques in 1955 but also contains additional information compiled in subsequent months.

Sommaire

DESCRIPTION D'UN JOINT ÉPROUVÉ et d'application étendue capable de tenir des vides trés élevés. Ce joint peut être facilement monté ou démonté. Il peut supporter des étuvages prolongés aussi bien qui des températures telles qui celle de l'azote liquide. Des joints ont été fabriqués leurs diamètres variant entre un et douze inches environ. Différents matériaux ont été joints.

Cet article est basé sur une conférence faite par les auteurs lors du Deuxième Symposium du Comité des Techniques du Vide en 1955. Cependant des informations supplementaires obtenues pendant les mois suivants y ont été ajoutées.

INTRODUCTION

THE IDEAL VACUUM SEAL must be able to contain an ultra-high vacuum (10^{-9}mm. Hg or less) without appreciable leakage, real or virtual, over extended time intervals. It must withstand thermal cycling and be both reliable and mechanically strong. The ideal seal is readily able to join many materials differing in physical properties in a wide range of sizes. Seal assembly should be simple and positive. Seal components should be re-usable and possess a low unit cost. The knife-edge vacuum seal, to be described, satisfies all of these requirements whereas the conventional techniques do not.

Organic seals range from O–rings to greases. They are of limited applicability because of the high vapour pressures (about 10^{-6} mm. at best) and poor thermal properties of suitable organics. They are moderately strong and can readily be assembled, joining a variety of materials in a range of sizes. These seals are best used in dynamic vacuum systems.

Glass-to-glass and glass-to-metal seals are of extreme importance in all vacuum work. Nevertheless, they possess a number of limitations. Reliability is not of the best, particularly for the larger joints assembled *in situ* and flame annealed. Skilled personnel and special equipment are required. These seals are re-usable a limited number of times, if at all. Hence, the cost is high. Only a limited number of materials in a restricted size range can readily be employed in such seals.

Metal-to-metal joints fall into two categories: Permanent, assembled by soldering, brazing or welding; and demountable, utilizing threads or metallic gaskets. The permanent metal seals can contain a

* MS. received November, 1956.

high vacuum when given a suitable alloy and cleaning. Apart from the soldered joint, they can withstand thermal cycling. They are strong and can handle a wide variety of sizes but only a limited number of materials. They can present serious problems in assembly. They can be a source of contamination because of the heat used, high vapour pressure components, fluxes, and gas pockets. With intermediate machining, the components are re-usable in general. These demountable metal joints depending on threads or the conventional flat metal gasket have no place in high vacuum work. The gasket-type vacuum closures, described in the literature, can be divided into four categories:

(1) metal O-rings,
(2) fine wire gaskets,
(3) cold-flow type closures,
(4) flat metal gaskets.

Many of these seals are characterised by their complexity with its associated high cost, limited flexibility or application, and poor exhaust characteristics. Insofar as flat metal gasket seals are concerned, the work of Arnett and Kyser[1], Pattee[2], and van Heerden[3] should be mentioned. The principle of these seals is similar to ours, differing primarily in the shape and finish of the knife edges and the mass of the parts.

DESIGN OF THE SEAL

The knife-edge vacuum seal is a demountable gasket type of seal, as shown in Fig. 1. The seal is made by forcing two mating knife edges to bite into a metallic gasket by the application of a suitable force. If the system is properly designed, the resulting joint is tight. To date, our experience is based only on radially symmetrical seals for the practical reason that they are by far the easiest to machine by using conventional shop techniques. Apart from the machining problem, there is no reason why other geometries could not be used. Ultrasonic shaping, electroforming, electromachining, or abrasive drill offer interesting possibilities for odd shapes.

This type of vacuum closure has proved most satisfactory. It has contained vacua of approximately 10^{-10} mm. Hg for extended times. Contamination is no problem. Failures are generally traced to bulb defects. These seals have been used in liquid nitrogen traps as well as for joints that are cycled repeatedly between 25 and 425°C. Faulty closures are few and readily corrected. Seals ranging from one-half to twelve inches in diameter have been constructed. Materials joined include ceramics, glass, metal, quartz, and sapphire. These seals are inexpensive, being easy to machine and assemble, and they can be re-used indefinitely.

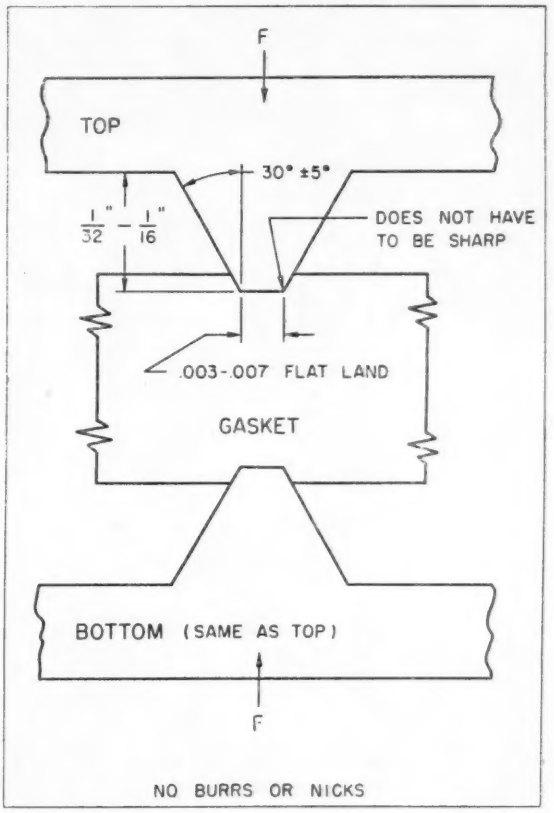

Fig. 1. Knife edge detail.

The details of the knife edge are shown in Fig. 1. Five points to be noted are:

(1) The land must be planar.
(2) The diameter of mating knife edges must be the same.
(3) The knife edges must be in line within 0.010 inch.
(4) The knife edge must be free of burrs or nicks.
(5) The force must be applied uniformly and normal to the knife edge.

The same profile and dimensions are used regardless of the seal diameter. The purpose of the land is to simplify the problem of generating a planar surface

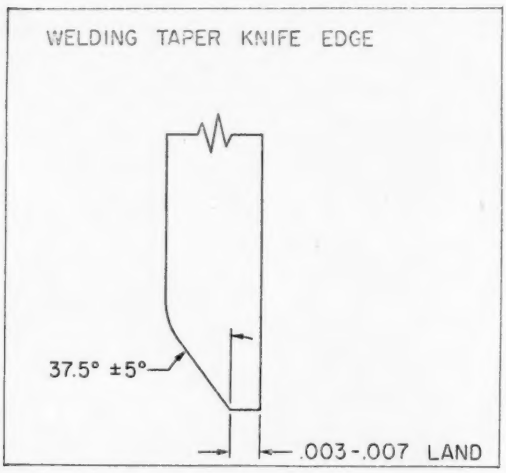

Fig. 2. Standard ASA B16.5 taper.

of contact. Actually, the width of the land can range from 0.003 inch to an extensive flat. The wider the land, the greater the force required for a satisfactory seal.

Our practice is to machine the knife edge completely either on the lathe or by lapping in a drill press. Either method produces a satisfactory surface. If the knife edge should be damaged, a simple lapping will correct it. Laps can be machined from copper or cast iron or formed by spinning or pressing the knife edge into a suitable plastic. For the larger knife edges, a lap can be moulded from those plastics which set in air, such as 'Devcon'. If the edge will be subject to considerable abuse, one can machine a ridge somewhat larger than needed and lap it to shape immediately before use. Two to three hundred mesh alumina, boron carbide, diamond grit or silicon carbide are suitable. Diamond grit and an acrylic plastic are universally satisfactory and free of objectionable materials.

The knife edge detailed in Fig. 2 has been used for the larger joints, since it requires but a suitable land machined on a standard welding taper. Our experience with this taper has not been satisfactory for the smaller joints because of alignment problems, and has not been investigated further.

Insofar as clamping is concerned, the details depend largely upon the particular set-up. In the examples to be presented later, the screw thread is used to generate the necessary pressure. Other clamping devices can be used readily, depending on the requirements of the set-up and the ingenuity of the experimenter. The sealing force should be generated by a device in stress, but not beyond its elastic limit, so that minor dimensional changes will be absorbed. This force is small considering standard 150–pound practices, as Table I illustrates. The compressive force must be uniformly applied and the use of appropriate bolt-tightening sequences is mandatory. If the bolt holes do not align the knife edges properly, guide devices must be used.

Table I. **Bolts Required**

Seal Diameter inch.	*Number of Bolts*	*Thread*
1	3	10/32
2	6	10/32
4	8	3/8
8	8	5/8
12	12	3/4

The knife edge must be of a harder material than the gasket. The harder and tougher the knife edge, the better, since those characteristics minimise the danger of nicking or burring, which can destroy the tightness of the seal. The use of carburising to increase the surface hardness is well worth considering. Table II lists knife-edge, flange, and gasket materials that have been investigated to date. In selecting a knife-edge material, some thought must be given to

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the rest of the system or failures will occur. For example, the 1.5–inch seal joins glass *via* a short length of 0.030–inch thick Kovar tubing to a knife edge. If the knife edge is of 304 stainless steel, the Kovar tubing should be longer than for a 4130 alloy ($\frac{1}{2}$ inch minimum, $\frac{3}{4}$ inch preferred).

Commercial sheet copper is suitable for gaskets. OFHC copper is not required. The gaskets are thin, $\frac{1}{16}$ inch stock being ample. Suitable copper washers can be ordered from a commercial supplier, specifying dead soft copper free from scratches. Odd sizes can be cut from sheet stock. The gaskets can be used as they are although, if desired, the gaskets can be air-fired to anneal, and wire-brushed or chemically cleaned to remove the resulting oxides and reduce any scratches. Circular scratches are of no consequence but axial ones may cause leaks. Another satisfactory method is to subject these gaskets to high pressures (50,000 psi) which results in a mirror-finish surface. At times, the gaskets are used to locate the knife edges. Such gaskets are machined out of much heavier stock. Appropriate shoulders serve as locating devices and the gaskets are re-usable by taking a light surface cut to eliminate the bite. The depth of bite into the gasket is small (from 0.005 inch to 0.020 inch), depending primarily upon the condition of the gasket surface.

Gaskets can be re-used several times if desired, but it becomes progressively difficult to accomplish the seal. The copper gaskets can take extended bake-outs without any ill effects. A limited number of soft aluminium gaskets were used as they were, also a number of 0.050–inch grade A nickel gaskets. Because of the resistance to oxidation, the nickel gaskets are of particular interest for seals which are kept at extended temperatures for prolonged periods of time. Noble metal gaskets could be used but are expensive. An-

Table II. **Seal Materials**

Knife-Edge	
Low Carbon Steel 4130 Alloy Steel 304 Stainless Steel Kovar Ceramics	Quartz Sapphire Carballoy* Glass
Gaskets	*Flanges*
Copper-Commercial, Dead Soft Aluminium-Commercial, Soft Nickel Grade A	Low Carbon Steel 304 Stainless Steel

* Tests Incomplete.

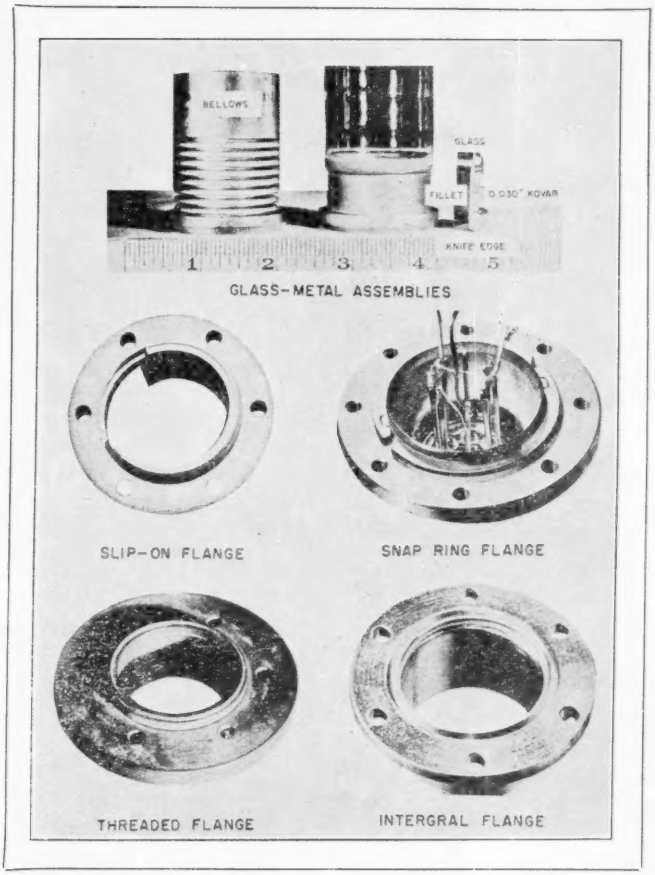


Fig. 3. The 1.5–inch knife-edge seal.

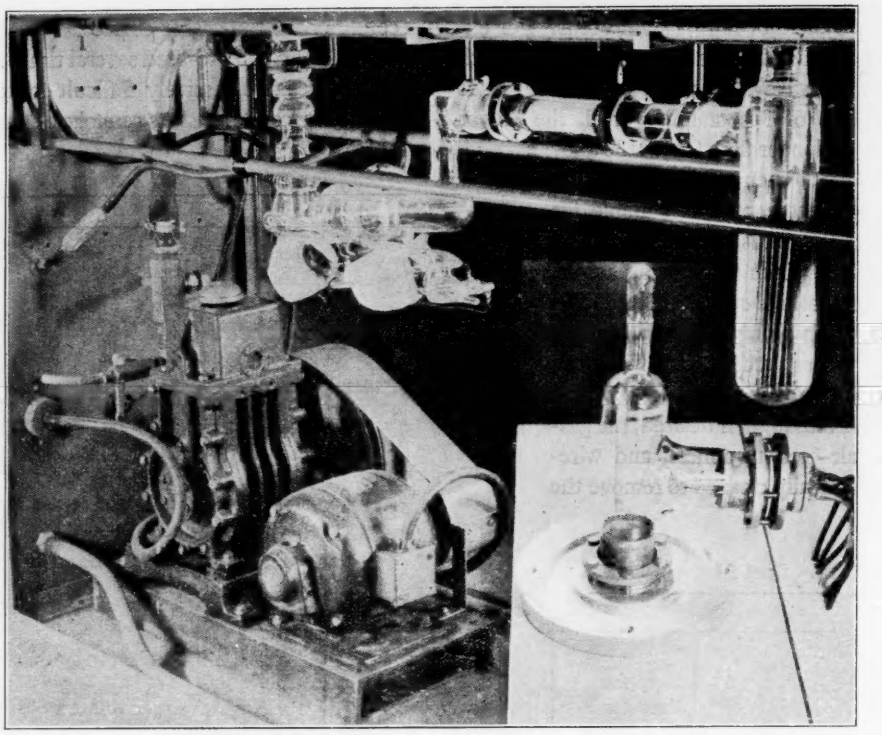
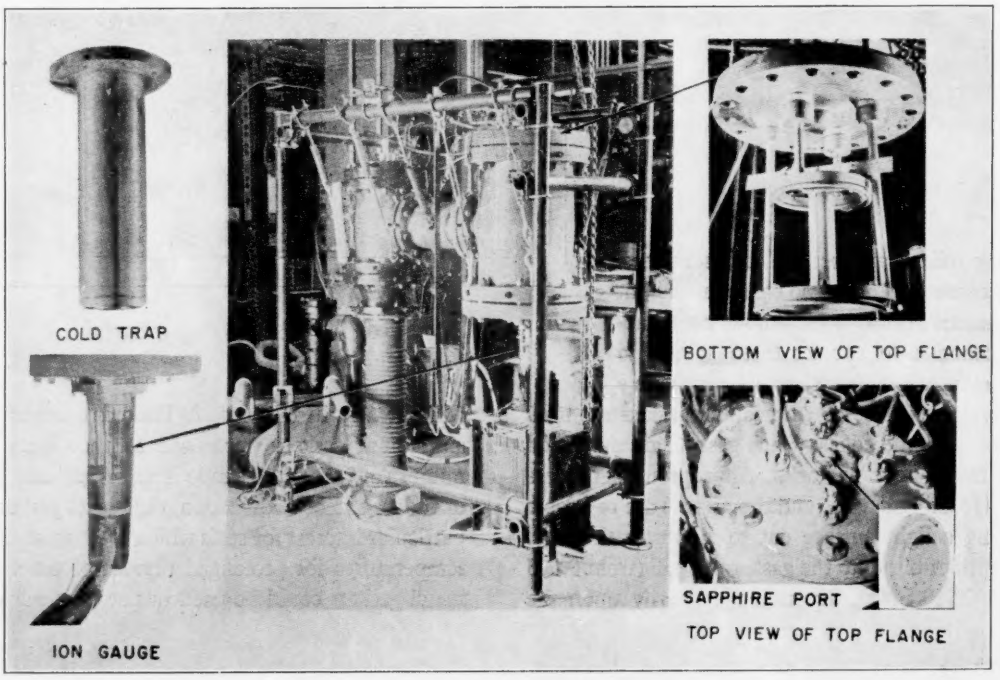


Fig. 4. (right) Modular vacuum system.

Fig. 5. (below) The high-vacuum, high temperature furnace.



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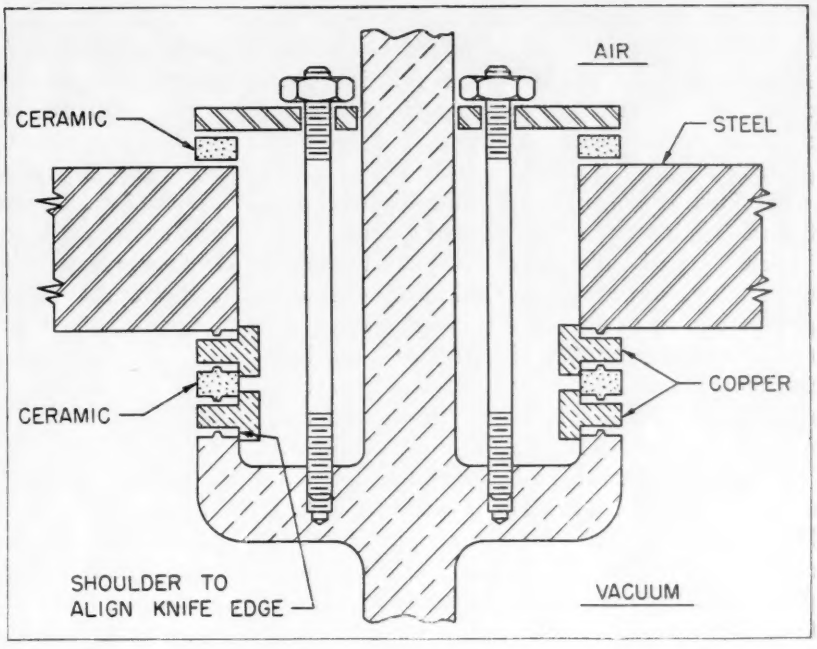

Fig. 6. Details of ceramic knife-edge insulated seal.

other solution might be to electroplate a copper gasket heavily with a noble metal that is then fused in place. Since there is no oxidation in the bite, this procedure should prove quite satisfactory.

The following photographs illustrate several applications of the knife-edge seal. All of these have satisfactorily contained pressures of 5×10^{-7} mm. or less.

Fig. 3 presents a few modifications of the 1.5–inch seal. This seal was primarily developed to join 1.5–inch glass to glass. Two glass-metal assemblies are shown, differing only in that one contains a bellows section to minimise alignment and strain problems when several components must be assembled together. Preferentially, the metal parts are furnace-brazed in hydrogen, using either Nicoro or the Au–Cu eutectic and the Kovar is then glassed and the whole assembly oven-annealed. Glassed Kovar has also been satisfactorily r.f. brazed to the remainder of the assembly. Four versions of the flange knife-edge assembly are also shown. The slip-on flanges seat on a shoulder machined on the glass side of the knife edge. If the flange is to be removable, this imposes the limitation that the o.d. of the glass must be less than the i.d. of the central hole in the flange. To eliminate this undesirable restriction, the other versions were constructed. The threaded knife edge is expensive, since the threads must be accurately machined and they are subject to oxidation. The snap ring design uses a Kovar knife edge but the availability of heavy wall Kovar tubing is limited. The integral knife-edge flange is limited to rotation in steps of 60°. These seals have contained vacua of approximately 10^{-11} mm. Hg. They have withstood 40–hour bake-outs at 400°C. They have withstood immersion in methanol-dry ice (–80°C). Using a Housekeeper seal, they can withstand immersion in liquid nitrogen (–186°C). Kovar seals usually will fail this test since Kovar undergoes a gamma-alpha transition in this region. A 3.5–inch integral knife-edge seal, made of cold rolled steel and used on a vacuum bottle, has a record of more than 300 successful closures before the knife edge was inadvertently damaged and had to be relapped.

The 1.5–inch seals have been used to assemble static, all-glass vacuum systems, as shown in Fig. 4. By using a modular construction and standard modules, one has a very flexible vacuum system capable of the highest vacua which can be assembled and disassembled quickly and without the special talents of a glass blower. Individual modules can be replaced or cleaned at will. Mixed materials, such as glass and metals, can be used. Glass troubles are minimal since all glass work is done at the bench and is oven annealed.

Pressure is measured by means of an appropriate ionisation gauge attached to the furnace proper, as shown in the insert on Fig. 4. This technique not only allows one to change gauges readily but also eliminates the tubulation which tends to isolate the gauge from the system.

Fig. 5 illustrates a high-temperature, high-vacuum furnace. This furnace easily reaches a pressure of 10^{-7} mm. Hg with the load at 1,500°C brightness, and a wall temperature of 250°C. The high-vacuum plumbing is assembled completely with the aid of the knife-edge seal. The fore-vacuum plumbing uses conventional threaded pipe joints with glyptal as the lacquer. To date, leaks have occurred only in the oil drain plug and the threaded fore-pump plumbing. The large Tee contains the furnace proper and the small Tee, the cold trap. The upper flange of the large Tee supports the current-carrying leads and a sapphire viewing port; the bottom Tee, the loading port with its test leads and an ionisation gauge. The knife edge was machined into a sapphire optical flat near its periphery. No re-polishing of the flat was required. One pair of the heater leads is insulated from this flange by using ceramic knife edges. This seal is shown in more detail in Fig. 6. Notice in particular the use of shoulders on the thick copper gaskets to align the knife edges.

The small Tee contains a liquid nitrogen trap located above a freon-cooled baffle which is used to isolate the pumps from the furnace proper. The cold trap is of stainless steel. Attempts to fabricate this trap by brazing were unsuccessful and, therefore, a knife-edge construction was developed. This trap has been cycled repeatedly between –190°C and room temperatures with no failures.

CONCLUSIONS

The knife-edge seal provides a useful technique for securing vacuum-tight closures over a wide range of applications, ranging from demountable tubes to entire vacuum systems. These seals have been successfully applied to pressurised systems on a limited basis. To date, their application to joints considerably larger than those discussed here has not been investigated but there is no reason why they should not prove satisfactory.

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TWO-LAYER ANTI-REFLECTION COATINGS FOR GLASS IN THE NEAR INFRA-RED

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Summary

THREE TYPES OF two-layer anti-reflection coatings capable of producing zero reflectance on glass of index 1.51 are discussed. All three types consist of a high-index inner film and a low-index outer film and use the same low-index film material (MgF_2 with $n_1=1.38$) but various high-index coatings. The first design consisting of two quarter-wave films requires $n_2=1.7$ for producing zero reflectance on glass at 1μ wavelength. The reflectance minimum of such double quarter-wave coatings is extremely sensitive to thickness variation of the low-index film and very much less influenced by mismatching of the high-index coating. The second type of coating consists of a thin high-index ($t_2<\lambda/4$) and a thick low-index ($t_1>\lambda/4$) film. This combination can yield zero reflectance for all values of $n_2>1.7$. The third coating type uses one half-wave and one quarter-wave film. With coatings of this type two reflection minima which become zero for $n_2=1.9$ can be obtained. Experimental coatings of the three types were used for anti-reflecting glass in the near infra-red. The coatings were prepared by high vacuum evaporation of magnesium fluoride, silicon monoxide and cerium dioxide.

Sommaire

TROIS TYPES DE DÉPÔTS, formés de deux films Anti-Reflexion capables de produire une reflexion nulle sur verre d'indice 1.51, sont discutés. Ces trois types, sont constitués d'un premier film à indice élevé, recouvert d'un film à indice bas, employant le même materiau pour le film à indice bas (MgF_2 avec $n_1=1.38$) mais différents dépôts à indice élevé. Dans le premier modèle constitué par deux films d'un quart de longueur d'onde, n_2 doit être égal à 1.7 pour produire une reflexion nulle sur verre pour une longeur d'onde de 1μ. La reflexion minimale de tels dépots est extrèmement sensible aux variations de l'épaisseur du film à indice bas, mais est moins sensible à un manque d'uniformité du film à indice élevé. Le deuxième consiste en un film mince ($t_2<\lambda/4$) et d'un film épais à indice bas ($t_1>\lambda/4$). Cette combinaison permet d'obtenir une reflexion nulle pour toutes les valeurs de $n_2>1.7$. Le troisième est constitué de deux films $\lambda/2$ et $\lambda/4$. Avec des dépôts de ce type on peut obtenir deux minima de reflexion qui deviennent zero pour $n_2=1.9$. Des dépôts expérimentaux de ces trois differents types ont été employés pour verre 'anti-reflexion' dans la region de l'infra-rouge. Ces dépôts furent faits en évaporant sous vide MgF_2, SiO et CeO_2.

INTRODUCTION

THE REDUCTION OF undesirable reflections from windows and lenses by the applications of thin films has become common practice in the field of optics. The technique of evaporation in high vacuum as a means for producing films with precisely controlled properties and thicknesses has greatly aided this development. A reduction of reflection losses on glass can be obtained with quarter-wavelength thick single films of low-index materials. Such single films are most efficient and result in zero reflectance at the quarter-wave position if their refractive index is equal to the square root of the glass index ($n_1=\sqrt{n_g}$). For the most frequently used glasses with $n_g\sim1.5$ a film material with $n_1=1.23$ would make an ideal anti-reflection coating. Unfortunately, durable film materials with such low indices of refraction are not available. Today, magnesium fluoride (MgF_2) with $n_1=1.38$ is most frequently used for preparing anti-reflection coatings on glass. MgF_2 films are very durable and decrease the surface reflectances of an ordinary glass plate from 8% to 2.8%. To obtain more efficient durable anti-reflection coatings on glass, double or multi-layer films have to be used.

* MS. received January, 1957.

It is the purpose of this paper to discuss the theory and practical applications of two-layer anti-reflection coatings on glass for the near infra-red.

THEORY OF TWO-LAYER ANTI-REFLECTION COATINGS

General

The theory of two-layer films has been discussed extensively in the literature[1,2]. Some of the more or less neglected special cases will be treated here. The types of film combinations to be discussed are illustrated in Fig. 1.

The entire discussion will be restricted to cases where $n_0=1$, $n_1=1.38$ (MgF_2), and $n_g=1.51$, allowing only n_2 to vary in such a way that $n_2>n_g$.

Any film combination composed of two dielectric layers on a semi-infinite substrate has a reflectance given by:

$$R=\frac{r_1^2+r_2^2+r_3^2+r_1^2r_2^2r_3^2+2r_1r_2(1+r_3^2)\cos 2\phi+2r_2r_3(1+r_1^2)\cos 2\psi+2r_1r_3\cos 2(\phi+\psi)+2r_1r_2^2r_3\cos 2(\phi-\psi)}{1+r_1^2r_2^2+r_1^2r_3^2+r_2^2r_3^2+2r_1r_2(1+r_3^2)\cos 2\phi+2r_2r_3(1+r_1^2)\cos 2\psi+2r_1r_3\cos 2(\phi+\psi)+2r_1r_2^2r_3\cos 2(\phi-\psi)} \quad (1)$$

Where:

$$r_1=\frac{n_0-n_1}{n_0+n_1};\qquad r_2=\frac{n_1-n_2}{n_1+n_2};\qquad r_3=\frac{n_2-n_3}{n_2+n_3};$$

$$\phi=\frac{2\pi n_1d_1}{\lambda};\qquad \psi=\frac{2\pi n_2d_2}{\lambda};$$

n = index of refraction
d = physical film thickness
λ = wavelength in same units as d

This expression is quite cumbersome and can be solved only by approximate numerical methods.

Coatings Consisting of Two Quarter-Wave Films

(See Fig. 1-A.) If the two layers are restricted to be of equal optical thickness, Equ. 1 simplifies to:

$$R=\frac{r_1^2+r_2^2+r_3^2+r_1^2r_2^2r_3^2+2r_2(r_1+r_3)(1+r_1r_3)\cos 2\phi+2r_1r_3\cos 4\phi}{1+r_1^2r_2^2+r_1^2r_3^2+r_2^2r_3^2+2r_2(r_1+r_3)(1+r_1r_3)\cos 2\phi+2r_1r_3\cos 4\phi} \quad (2)$$

and if both films are one quarter wavelength thick on glass (n_g), Equ. 2 reduces to:

$$R=\left(\frac{n_1^2n_g-n_2^2}{n_1^2n_g+n_2^2}\right)^2$$

This shows immediately that zero reflectance can be obtained with a double quarter-wave coating if the indices of refraction satisfy the following relation:

$$\frac{n_2}{n_1}=\sqrt{n_g} \quad (3)$$

On glass of $n_g=1.51$ with an outer film of $n_1=1.38$ (magnesium fluoride), an index $n_2=1.7$ is, therefore, required for the inner film to produce zero reflectance at the quarter-wave position λ_0.

Fig. 2 compares the calculated reflectance of a single ($n_1=1.38$) and a double ($n_1=1.38$ and $n_2=1.7$) anti-reflection coating on glass with $n_g=1.51$. The double-layer coating results in zero reflectance but is superior to the single coating only over a rather narrow region.

Fig. 3 shows the minimum reflectance obtainable with the described two quarter-wave film when n_2 deviates from the value ($n_2=1.7$) which satisfies Equ. 3. Although the refractive index n_2 of the inner film should be 1.7 for strictly zero reflectance, from a practical point of view considerable deviation from the ideal value is often permissible. A variation by as much as 5 per cent. from the ideal value of 1.7 results in a reflectance rise at the minimum of only 0.25 per cent.

Fig. 4 demonstrates the effect of thickness mismatching on the reflectance minimum of double-layer coatings with $n_1=1.38$ and $n_2=1.7$ on glass of $n_g=1.51$. It is at once evident that the reflectance minimum is extremely sensitive to thickness variations of the low-index film t_1, but that a thickness variation of the high-index film has very little effect on the reflectance minimum. The deposition of a uniform, truly one quarter-wavelength thick, low-index film is, therefore, most important for the preparation of an

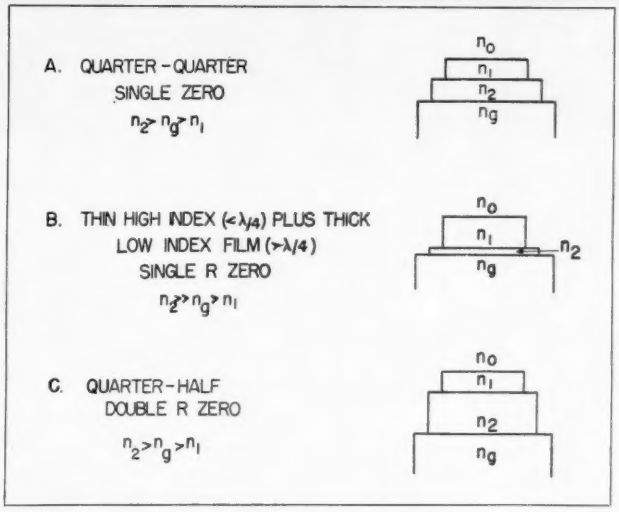

Fig. 1. Types of two-layer anti-reflection coatings discussed in this paper.

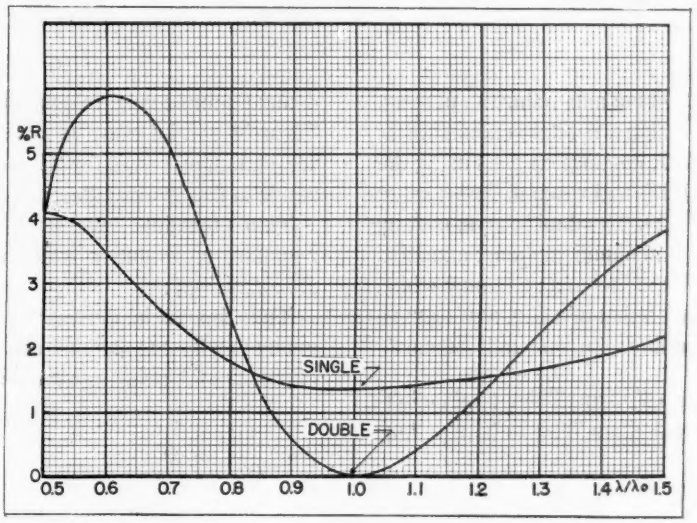

Fig. 2. Calculated reflectance of one glass surface ($n_g = 1.51$) coated with single ($n_1 = 1.38$) and double ($n_1 = 1.38$, $n_2 = 1.7$) quarter-wave anti-reflection coatings as function of λ/λ_0.

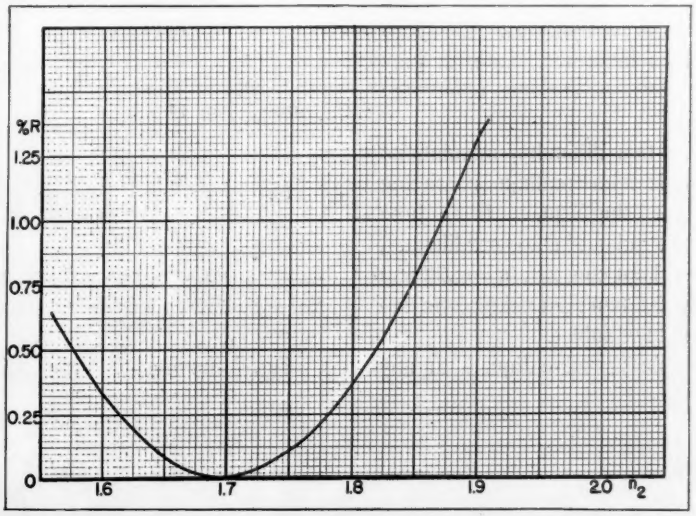

Fig. 3. Reflectance minimum as function of n_2 for double quarter-wave coatings on glass with $n_g = 1.51$ and outer film index $n_1 = 1.38$.

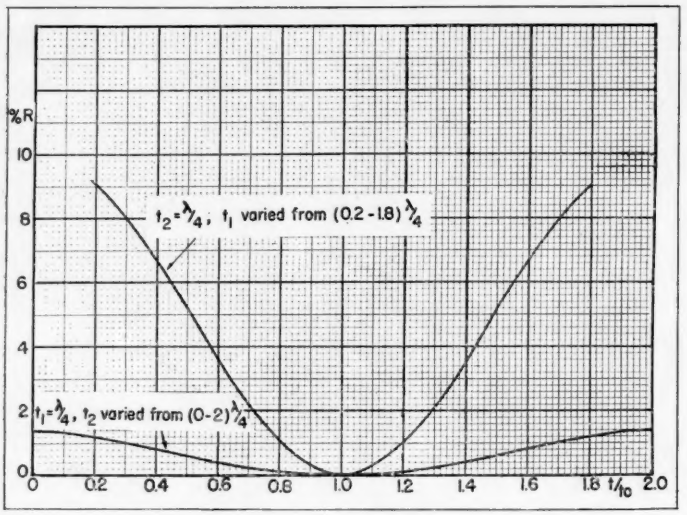

Fig. 4. Effect of thickness mismatching on the reflectance minimum of double-layer coatings with $n_1=1.38$ and $n_2=1.7$ on glass with $n_g=1.51$. Optical thickness $t_0=\lambda/4$.)

effective double quarter-wave anti-reflection coating.

Fig. 5 shows the calculated spectral reflectance of three double quarter-wave coatings on glass using the same outer film ($n_1=1.38$) and three different inner films ($n_2=1.6, 1.7$, and 1.8). The double-layer coating with $n_1=1.38$ and $n_2=1.6$ shows the most extended low reflection region and a reflectance minimum which is only slightly higher than that of a film combination satisfying the index condition for zero reflectance.

Coatings Consisting of One Thin ($n_2d_2<\lambda/4$) High-Index Film and One Thick ($n_1d_1>\lambda/4$) Low-Index Film

(See Fig. 1–B). In the discussion of double quarter-wave coatings it was shown that zero reflectance can only be obtained with films satisfying the index conditions of Equ. 3. This limitation makes it very difficult in practice to obtain a true zero reflectance with quarter-wave films.

In order to surmount this difficulty, calculations were extended to double-layer coatings consisting of non-quarter-wave films using Equ. 1. In all these calculations the refractive index of the outer film, n_1, and of the glass, n_g, were kept constant ($n_1=1.38$ and $n_g=1.51$), leaving only the two film thicknesses expressed by ϕ, ψ, and n_2 as variables.

Since it is not possible to solve Equ. 1 in any direct algebraic way, values have to be assigned to n_2. Then 2ϕ and 2ψ must be varied simultaneously until the particular values for 2ϕ and 2ψ are found which result in the lowest reflectance value.

This procedure involves a large amount of unnecessary computation which can be reduced in the following way:

Take the partial derivatives of R (in Equ. 1) with respect to 2ϕ and 2ψ and set them equal to zero to obtain the following equations:

$$from \frac{\partial R}{\partial 2\phi} = O$$

$$tan\ 2\phi = \frac{-r_3(1-r_2^2)\ sin\ 2\psi}{r_2(1+r_3^2)+r_3(1+r_2^2)\ cos\ 2\psi} \qquad (4)$$

$$from \frac{\partial R}{\partial 2\psi} = O$$

$$tan\ 2\psi = \frac{-r_1(1-r_2^2)\ sin\ 2\phi}{r_2(1+r_1^2)+r_1(1+r_2^2)\ cos\ 2\phi} \qquad (5)$$

Then a plot of 2ϕ as a function of 2ψ is made with these equations using given indices for calculating r_1, r_2 and r_3.

Fig. 6 shows such a plot using $n_1=1.38$, $n_2=2.6$,

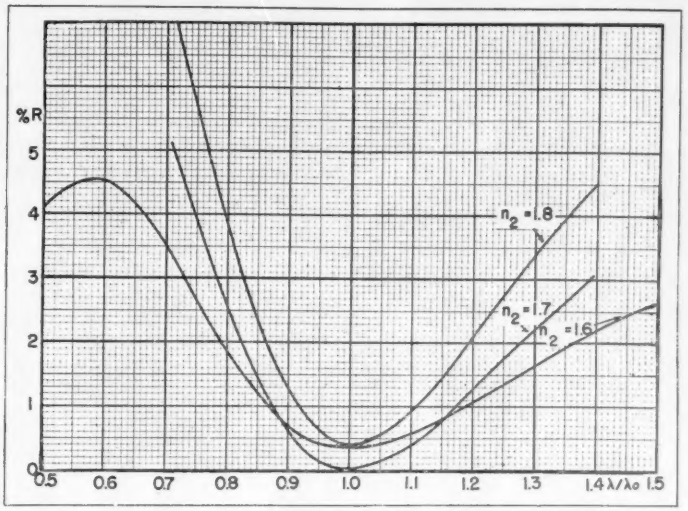

Fig. 5. Calculated spectral reflectance of one glass surface ($n_g = 1.51$) for three double quarter-wave coatings using the same outer film ($n_1 = 1.38$) and three different inner films ($n_2 = 1.6$, 1.7 and 1.8).

and $n_g = 1.51$. The points of intersection represent simultaneous solutions corresponding to maxima or minima of reflectance. Most of the reflectance maxima and minima occur for values of 2ϕ and 2ψ which are integral multiples of π (corresponding to film thicknesses of integral multiples of $\lambda/4$). Film combinations of this type can be calculated rather easily and have been studied extensively in the literature. The two most important examples of this group are the double quarter-wave coatings already discussed in the previous section and the quarter-wave-half-wave combination to be discussed in the following section.

There are, however, other simultaneous solutions for which 2ϕ and 2ψ are not integral multiples of π or 180°. These solutions which correspond to non-quarter-wave film combinations have received very little attention in the literature. All of these intersections shown in Fig. 6 represent minima with zero reflectance.

The reflectance minima to be discussed in this section occur at values of 2ϕ between π and 2π (t_1 between $\lambda/4$ and $\lambda/2$) and of 2ψ between 0 and π (t_1 between 0 and $\lambda/4$). To find the thicknesses of t_1 and t_2 resulting in zero reflectance for various film combinations, plots of Equs. 4 and 5 were made using the same $n_1 = 1.38$ and $n_g = 1.51$, but different n_2. From the points of intersection, t_1 and t_2 were determined and used in Equ. 1 to calculate the spectral reflectance of the double-layer coating.

Fig. 7 shows the plots of Equs. 4 and 5 for two double-layer coatings on $n_g = 1.51$. The first coating consists of films with $n_1 = 1.38$ and $n_2 = 1.8$, and shows zero reflectance for $2\phi = 212.5°$ ($t_1 = 1.18 \times \lambda/4$) and

Table 1. Film Thicknesses in Double-Layer Coatings Producing Zero Reflectance on Glass with $n_g = 1.51$.

Various High-Index Films ($n_2 = 1.7$ to 3.0) Combined with Same Low-Index ($n_1 = 1.38$)

n_2	t_2 Degrees	t_2 $\lambda/4$	n_1	t_1 Degrees	t_1 $\lambda/4$
1.7	90.0	1.00	1.38	90.0	1.00
1.8	45.5	0.51	1.38	106.2	1.18
1.9	31.5	0.35	1.38	110.7	1.23
2.0	26.0	0.29	1.38	113.6	1.26
2.2	19.5	0.21	1.38	116.3	1.29
2.4	15.8	0.18	1.38	117.8	1.31
2.6	14.5	0.16	1.38	118.7	1.32
2.8	12.6	0.14	1.38	119.5	1.33
3.0	11.2	0.12	1.38	119.9	1.33

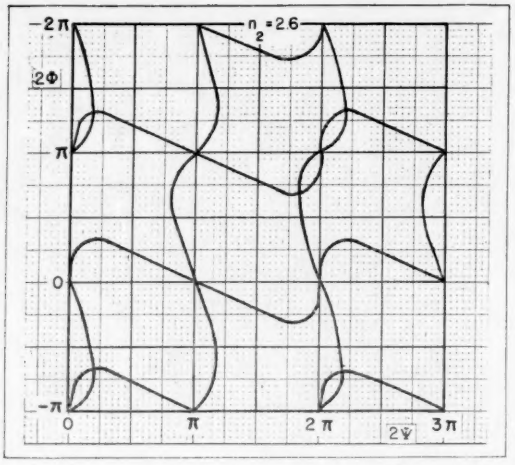

Fig. 6. 2σ as a function of 2ψ from Equs. 4 and 5, using $n_1=1.38$, $n_2=2.6$ and $n_g=1.51$ for calculating r_1, r_2 and r_3.

$2\psi=91°$ ($t_1=0.51\times\lambda/4$). The second coating with $n_1=1.38$ and $n_2=2.6$ results in zero reflectance for $2\phi=237.4°$ ($t_1=1.32\times\lambda/4$) and $2\psi=29°$ ($t_2=0.16\times\lambda/4$).

While the double quarter-wave coating with $n_1=1.38$ gives zero reflectance on glass ($n_g=1.51$) only for $n_2=1.7$, the discussed non-quarter-wave combinations yield zero reflectance for all values of $n_2>1.7$.

Table 1 lists the film thicknesses of double-layer film combinations which give zero reflectance on glass with $n_g=1.51$. All film combinations use the same $n_1=1.38$ but various values for n_2 ($n_2=1.7$ to 3.0). For convenience, the optical film thicknesses are given both in degrees and in quarter-wave units. As the refractive index of the high-index film n_2 increases, t_2 becomes increasingly smaller and t_1 larger than $\lambda/4$.

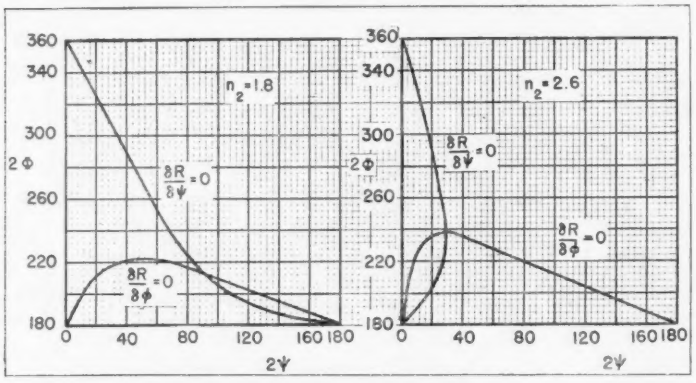

Fig. 7. 2σ as a function of 2ψ from Equs. 4 and 5 for two double-layer film combinations with $n_1=1.38$, $n_2=1.8$, and $n_1=1.38$, $n_2=2.6$ on glass with $n_g=1.51$.

An approximate vector method using the amplitude reflectances given by the Fresnel coefficients may also be used to obtain values for t_1 and t_2. As long as the film indices are low the accuracy of this method is quite good, but with increasing film indices the results deviate more and more from the correct values.

For the preparation of the double-layer coatings it is necessary to know the reflectance of the high-index film alone on glass so that its thickness can be controlled during the deposition. Fig. 8 shows the reflectance of one glass surface coated with a film of thickness t_2 as shown in Table 1. It is very convenient that the required high-index film increases the glass reflectance by nearly the same amount (4% to 7.5%) for all $n_2>2.1$, and that only a slightly higher reflectance increase must be produced if films with lower indices are used.

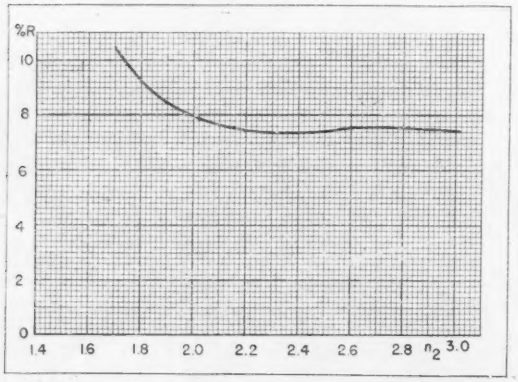

Fig. 8. Reflectance of one glass surface ($n_g=1.51$) coated with t_2 of Table 1.

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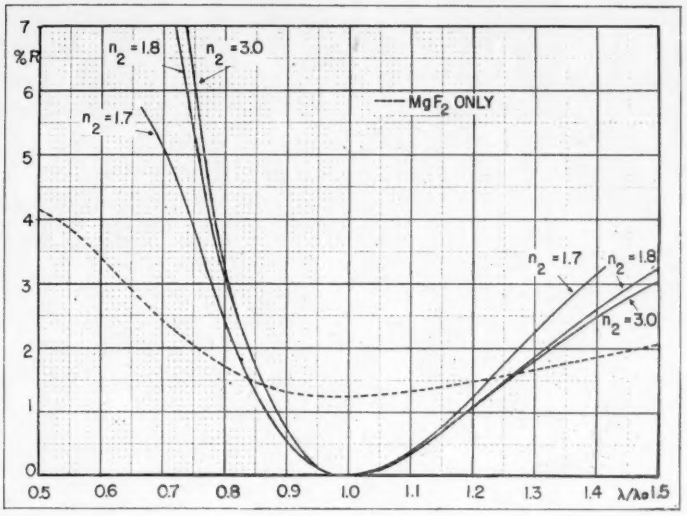

Fig. 9. Spectral reflectance of one glass surface ($n_g = 1.51$) for three double-layer coatings using the same outer film material ($n_1 = 1.38$) and three different inner films ($n_2 = 1.7$, 1.8 and 3.0) calculated with t_1 and t_2 of Table 1.

Fig. 9 shows the spectral reflectance of various non-quarter-wave anti-reflectance coatings on glass. The curves were calculated from Equ. 1 using t_1 and t_2 of Table 1. With increasing n_2 the reflectance rise is steeper at the shorter and slower at the longer wavelength side of the reflectance zero.

The reflectance minima of the coatings shown in Fig. 8 are again very sensitive to mismatching of the low-index films and very much less influenced by variations of the high-index coatings.

Coatings Consisting of One Half-Wave and One Quarter-Wave Film

(See Fig. 1–C.) To extend the low reflection region

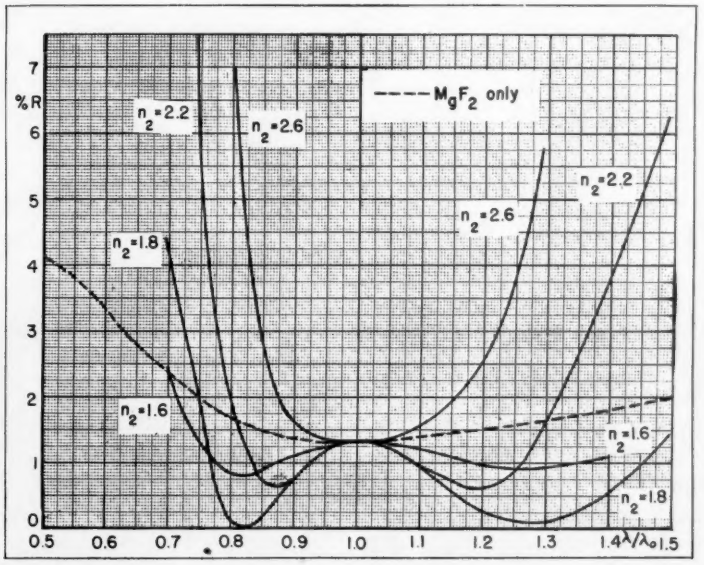

Fig. 10. Calculated spectral reflectance of one glass surface ($n_g = 1.51$) coated with several half-wave quarter-wave coatings.

All coatings use same index for quarter-wave film ($n_1 = 1.38$) but different indices for half-wave coating ($n_2 = 1.6$, 1.8, 2.2 and 2.6).

with double-layer coatings, half-wave plus quarter-wave films have to be used[2]. With coatings of this type, two reflection minima which may even become zero can be obtained. Fig. 10 shows the calculated reflectance of glass ($n_g=1.51$) coated with several half-wave quarter-wave film combinations. All combinations use the same index for the quarter-wave film ($n_1=1.38$) but various indices for the half-wave coating ($n_2=1.6$, 1.8, 2.2, and 2.6). The reflectance of glass coated with a single quarter-wave coating of $n_1=1.38$ is shown for comparison on the same graph. By increasing the refractive index of the half-wave film from 1.6 to 2.2 the reflectance of the two minima of the double-layer coating first decreases from 0.9% to zero at $n_2 \sim 1.9$ and then increases to 0.6%. The wavelength separation of the two minima decreases with increasing values of n_2.

If one side of glass is coated with a quarter-wave film of MgF_2 ($n_1=1.38$) and the other side with a half-wave quarter-wave coating of $n_1=1.38$ and $n_2=1.6$, uniform low reflectance of about 2.8 per cent. is obtained over a wavelength interval from (0.77–1.28) λ_0.

EXPERIMENTAL TECHNIQUES

All coatings discussed in this report were produced by evaporation in high vacuum. While other methods have also been successful for preparing anti-reflection coatings[3,4], the high vacuum evaporation techniques appeared to be most advantageous from the standpoint of ease, control, reproducibility, and variety of film materials available. The films were prepared in an 18-inch. vacuum chamber in which a pressure of 1×10^{-5} mm. Hg could be maintained throughout an evaporation. The vacuum unit was equipped with a glow discharge ring and heating elements to clean and heat the glasses to be coated. A built-in photometer was used to measure and control the film thicknesses during the film depositions. A removable Japanese fan-type shutter was provided between the evaporation sources and the substrate holder to avoid contamination of glass plates while the evaporation materials and sources were being outgassed. It was opened when the evaporation reached a constant speed and was closed when the film showed the desired thickness.

The film materials used for preparing various types of two-layer anti-reflection coatings were magnesium fluoride (MgF_2), silicon monoxide (SiO), and cerium dioxide (CeO_2).

Magnesium fluoride was evaporated from boats made of 0.008 inch thick sheet tantalum. The current required for the evaporation was about 300 A. To produce hard and durable coatings, the glass to be coated was heated to more than 300°C and the pressure was kept below 5×10^{-5} mm. Hg during the deposition. The refractive index of evaporated magnesium fluoride films was determined to be $n=1.38$ at $\lambda=1.0$ micron, which is identical with that of bulk magnesium fluoride and in good agreement with measurements made by Hall and Ferguson[5].

Silicon monoxide was evaporated from a directly heated tantalum boat or from a fused quartz crucible heated by a tungsten spiral placed over the silicon monoxide filled crucible. The refractive index of vacuum deposited silicon monoxide was found to vary considerably with the evaporation conditions[6]. Reduced silicon monoxide with a refractive index $n>1.9$ at 1 micron can be produced by rapid evaporation at low pressure ($p\sim1$ to 2×10^{-5} mm. Hg); oxidised films with an index $n<1.8$ are deposited by slow evaporation at higher pressures ($p\sim1$ to 2×10^{-4} mm. Hg). For the preparation of high-index films, evaporation from a tantalum boat was preferred while low-index films were deposited from silicon monoxide-filled, indirectly heated, fused quartz crucibles. Silicon monoxide was deposited on glass plates heated to more than 200°C since heating improved the adherence and hardness of the condensed films.

Cerium dioxide was evaporated from 0.012 inch thick tungsten boats. The refractive index and durability of evaporated cerium dioxide films were found to depend strongly on the temperature of the glass on which they were condensed[7]. An increase of the substrate temperature from 25°C to 300°C increases the refractive index of the condensed film material at $\lambda=1.0$ micron from about 2.15 to 2.35. A substrate temperature of more than 150°C is necessary to produce extremely hard and durable films.

The reflectance and transmittance of the coated

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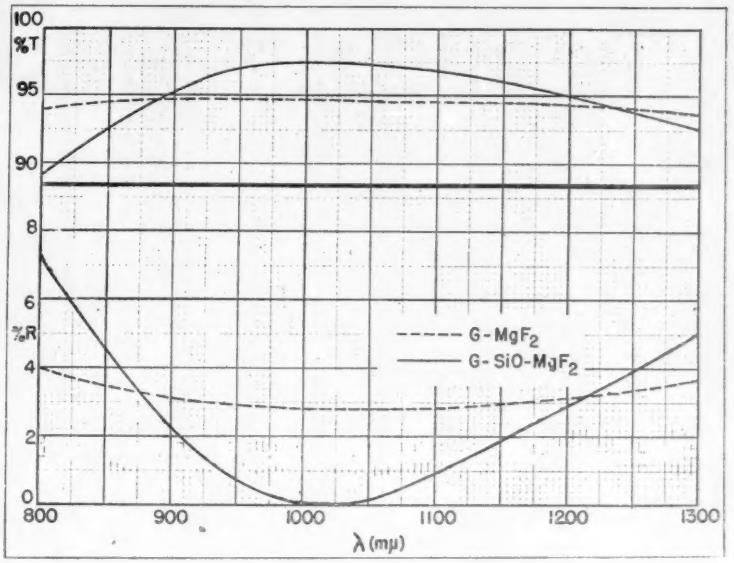


Fig. 11. Measured reflectance and transmittance of glass plates ($n_g = 1.51$) coated with single (MgF_2) and double quarter-wave ($SiO + MgF_2$) films for lowest reflectance at $\lambda = 1{,}000$ mμ.

glass plates were measured from 0.6 to 2.7 micron with a modified Beckman Model DU spectrophotometer and with a Perkin-Elmer Model 21 double-beam recording spectrophotometer.

RESULTS AND DISCUSSION

Coatings Consisting of Two Quarter-Wave Films

A single reflectance zero from glass can be obtained with a double quarter-wave coating satisfying the index condition:

$$\frac{n_2}{n_1} = \sqrt{n_g}$$

On glass of $n_g = 1.51$ with magnesium fluoride ($n_1 = 1.38$) as outer film an index $n_2 = 1.70$ is required for the inner film. Slow evaporated silicon monoxide meets this index requirement and is non-absorbing in the infra-red up to 8 micron. It is, therefore, especially suitable for preparing double quarter-wave anti-reflection coatings with magnesium fluoride on glass for the near infra-red.

To produce double quarter-wave anti-reflection coatings, the glass was first heated to about 250°C and silicon monoxide was deposited slowly (deposition rate ~ 3 to 4 Å/sec.) at a pressure of 1 to 2×10^{-4} mm. Hg until the surface reflectance reached a maximum. The substrate was then heated to more than 300°C and coated rapidly (25–30 Å/sec.) at a pressure $< 5 \times 10^{-5}$ mm. Hg with magnesium fluoride until the surface reflectance reached a minimum. With this procedure it was possible to reduce the glass surface reflectance at the control wavelength to practically zero. Fig. 11 and 12 show the reflectance and transmittance curves of two glass plates coated in this manner for minimum reflectance at 1.0 and 2.2 micron. For comparison, the reflectance and transmittance curves of glass plates coated with single films of magnesium fluoride for minimum reflectance at the

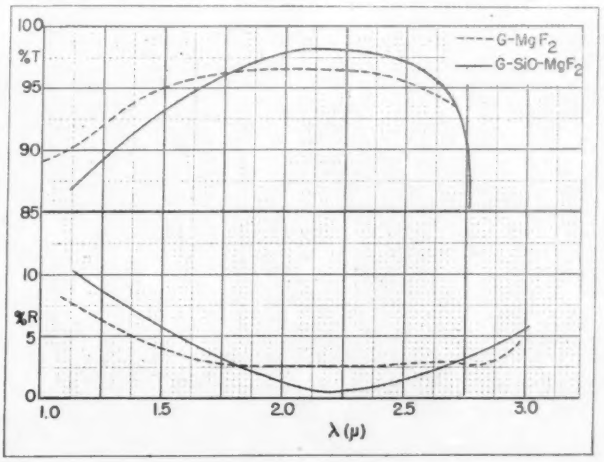


Fig. 12. Measured reflectance and transmittance of glass plates ($n_g = 1.51$) coated with single (MgF_2) and double quarter-wave ($SiO + MgF_2$) films for lowest reflectance at $\lambda = 2.2\mu$.

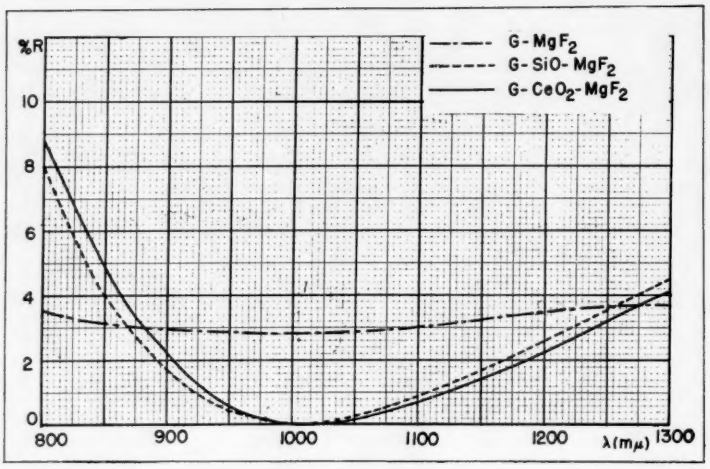
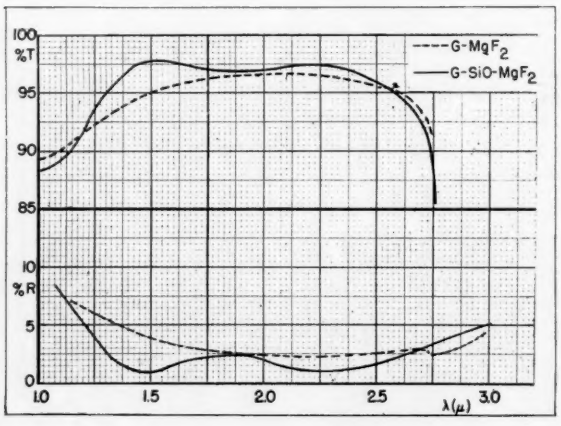


Fig. 13. Measured reflectance of glass plates ($n_g = 1.51$) coated with single (MgF_2) and double non-quarter-wave films of $CeO_2 + MgF_2$ and $SiO + MgF_2$ for lowest reflectance at $\lambda = 1{,}000$ mμ.

same wavelengths are plotted in the same Figures. The glasses used as substrates for all the coatings were 2 mm thick and showed an absorption of 2.5% at 1 micron and 2.0% at 2.2 micron. Consequently, the maximum transmittance with a coating which reduced the surface reflectance to near zero was 97.5% at 1 micron and 98% at 2.2 micron. The minimum reflectance of a two quarter-wave coating is up to 2.8% lower than that of a single quarter-wave film of magnesium fluoride. However, the two-layer coating is superior only over a rather narrow spectral region (from 0.85 to 1.2 micron for the 1–micron coating, and from 1.8 to 2.7 micron for the 2.2–micron coating).



Whether the reduction in reflectance, or the gain in transmittance, over such a restricted wavelength interval by the two two-layer coatings is significant in practice will depend on the problem to be treated. If the wavelength interval of interest covers a wavelength range not greater than $(0.85 \text{ to } 1.2) \times \lambda_0$, and if a great number of lenses and windows are present in the equipment, the two quarter-wave coatings will offer sufficient improvement to warrant their use. For some of the near infra-red viewing devices which have an effective wavelength range from 0.9 to 1.2 micron, two-layer coatings can be quite useful.

The two-layer coatings of silicon monoxide and magnesium fluoride were tested for abrasion resistance and hardness by rubbing with a pencil eraser and by scratching with a wire paper-clip and remained unmarked after these tests. They also showed no damage and no change in reflectance after a 24–hour salt spray test and two weeks constant immersion in a 5% sodium chloride solution.

Coatings Consisting of Two Non-Quarter-Wave Films

It was shown theoretically that zero reflectance can be obtained on glass with many two-layer film combinations consisting of high-index films thinner than a quarter wavelength and low-index films thicker than a quarter wavelength. On glass with $n=1.51$ and magnesium fluoride ($n=1.38$) as outer layer, zero reflectance can be obtained in combination with all inner film materials having $n > 1.7$. Experimental coatings of this type were produced with magnesium

Fig. 14. Measured reflectance and transmittance of glass plates ($n_g = 1.51$) coated with single films of $\lambda/4$ MgF_2 and with double layers of $\lambda/2$ $SiO + \lambda/4$ MgF_2 for lowest reflectance at the 2–micron region.

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fluoride as low-index material and cerium dioxide (n=2.2–2.3) and fast evaporated SiO ($n \sim 1.9$) as high-index materials. Fig. 13 shows the reflectance of glass plates coated with MgF_2–SiO and MgF_2–CeO_2 films for minimum reflectance at 1,000 millimicron. Both silicon monoxide and cerium dioxide were deposited with a deposition rate of 10–15 Å/sec. at $p \sim 1 \times 10^{-5}$ mm. Hg until the surface reflectance controlled at 1 micron increased from 4% to 8% or 7.5% respectively. Then magnesium fluoride was condensed until the reflectance reached a minimum. The reflectance characteristics of the double non-quarter-wave coatings are very similar to those of the double quarter-wave coatings (Fig. 11). The non-quarter-wave coatings show only a slightly steeper reflectance rise at the shorter wavelength side of the reflectance minimum and a slower rise at the long wavelength side than the quarter-quarter films. The wavelength range, in which both types of double-layer coatings are more effective than single films of magnesium fluoride, is almost the same.

The abrasion and corrosion resistance of CeO_2-MgF_2 coatings was found to be as good as that of SiO–MgF_2 films if the coatings were applied to heated substrates (>200°C for cerium dioxide and >300°C for magnesium fluoride).

In choosing between the described two types of two-layer film combinations most of the factors seem to favour the double non-quarter-wave coating, because it is not sensitive to variation in index of the high-index film and thus avoids the problem of regulating pressure and deposition rate required for producing silicon monoxide films with n=1.7. For anti-reflection coatings in the visible region, where silicon monoxide shows more or less absorption, double non-quarter-wave coatings of CeO_2–MgF_2 are more suitable.

Coatings Consisting of a Half-Wave and a Quarter-Wave Film

An approach to the problem of anti-reflecting glass over an extended spectral range is afforded by a double coating consisting of a half-wavelength-thick high-index film adjacent to glass and a quarter-wavelength-thick low-index coating as outer film. (Calculated curves of this type were presented in the theoretical part of this paper.) Fig. 14 shows the reflectance and transmittance of glass (n_g=1.51) coated on both sides with a single layer of magnesium fluoride (dashed curves) and with half-wavelength-thick silicon monoxide ($n_2 \sim 1.7$) and quarter-wavelength-thick magnesium fluoride (solid curves). The preparation of the coatings was controlled at 1.85 micron. The double-layer coating furnishes reflectance minima of about 1% at 1.5 and 2.3 micron. Between both minima the reflectance rises to that of a single layer of magnesium fluoride. This type of double-layer coating is superior to a single quarter-wave film of magnesium fluoride over the wavelength interval from 1.2 to 2.6 micron. It could be improved still further by employing faster evaporated silicon monoxide with n_2=1.9. Then the two reflection minima would go to zero.

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PERFECTION OF CONTOUR BY VACUUM DEPOSITED THIN FILMS

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Summary

THE FIDELITY OF CONTOURING of surface micro-steps by evaporated thin deposits of silver, cryolite and zinc sulphide is studied by precision multiple-beam interferometry. As a known step a spiral on silicon carbide is used and successive layers of thin film are deposited on this, the step being re-measured for each increment of deposit. With silver the contour is exact to within plus or minus 3 Å up to an overlying silver thickness of some 27,000 Å, although the step height being examined is only 480 Å in height. Up to thicknesses of 6,000 Å the measured step is correct very close to the step edge but beyond this thickness a defect sets in, such that there is a build-up on the step riser which reaches a maximum of about 100 Å close to the edge. With cryolite and zinc sulphide these defects are notably more marked. The experiments establish that the type of silver coating used in interferometry can be considered to contour surfaces perfectly. Multilayers may tend to obliterate microstructure if this is on a very small scale in extension.

Sommaire

LA FIDÉLITÉ d'épouser les micro-dénivellations de surface par des dépôts minces d'argent, de cryolithe ou de sulfure de zinc, est étudiée avec précision au moyen d'un interferomètre à faisceau multiple. Comme dénivellation connue on utilise une spirale de carbone de silicium sur laquelle on dépose des couches successives de film mince, la dénivellation étant remesurée pour chaque augmentation de dépôt. Avec l'argent la fidélité est de l'ordre de plus ou moins 3Å jusqu'à une épaisseur maximum de 27,000Å environ, bien que la hauteur de la dénivellation examinée est seulement de 480Å. Jusqu'à une épaisseur de 5,000Å la dénivellation mesurée est correcte ceci trés près de la marche de la dénivellation, mais au-delà de cette épaisseur un défaut apparait, tel que l'on observe une augmentation d'épaisseur sur le bord de la marche, allant jusqu'à 100Å. Avec la cryolitte et le sulfure de zinc, ces effets sont notablement plus marqués. Les experiénces démontrent que le type de dépôt d'argent employé en interférométrie peut être considéré comme épousant parfaitement les surfaces. Des couches multiples peuvent tendre à supprimer les micro-structures, ceci sur une très petite échelle en extension.

INTRODUCTION

DURING RECENT YEARS extensive use has been made of the techniques of multiple-beam interferometry. These methods have been applied in particular to the study of the microtopography of surfaces, to the examination of the structure of thin films and to the direct measurement of the thickness of thin metal and dielectric film deposits. With the correct technique a precision can be obtained so high that it is indeed possible to resolve and measure dimensions of the order of crystal lattice spacings, (in the vertical direction only, of course). Such a degree of precision necessitates the use of a highly reflecting thin film of metal or a high-reflectivity dielectric multilayer deposited by vacuum evaporation on the surfaces involved. The metallic films which have been found best are those of silver, some 500 to 1,000 Å thick. The dielectric multilayers employed in measurements of this kind consist of alternate quarter-wave layers of zinc sulphide and cryolite, the number of layers varying from three to nine, according to the problem in hand.

* MS. received December, 1956.

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Much has been published on work using these methods and in all these publications it is implicitly assumed that the thin reflecting film or multilayer exactly contours the surface topography. There are, indeed, several indications supporting indirectly the correctness of this assumption. For example, when the values of the cleavage steps on a good piece of muscovite mica are determined by multiple-beam interferometric measurement, it is found that, in general, the steps represent an exact small integral multiple of 20 Å, and this quantity coincides with the X-ray value of the lattice spacing between the cleavage planes.

Experiments with the epitaxial growth of ammonium iodide on muscovite strongly suggests that between cleavage lines mica can be considered to be crystallographically true to a single lattice. This has in fact been proved interferometrically. Both, this condition and the further observation that a lattice step is found interferometrically to be often quite uniform over lengths even of a centimetre or more, are supporting evidence indicating perfection of contour.

To quote another instance, I first (independently) made optical interference monochromatic filters in 1942. My technique was as follows: I used quite crude glass (a piece of an old photographic plate) on which I deposited a layer of silver, then a layer of cryolite, and finally a top layer of silver. The colour transmission characteristics of such a filter are highly sensitive to the thickness of the cryolite layer. Indeed, a thickness variation of 80 Å will readily show its effect on the colour transmission. Yet crude glass with local variations of several wavelengths (tens of thousands of Ångstroem) can be employed and still give a very good filter, uniform and monochromatic. Clearly, the three successive layers are contouring the surface topography with such fidelity that the cryolite sandwich is of uniform thickness, at least to within 80 Å. This indicates high fidelity contouring.

Finally, in many complex microtopographic studies dimensions predicted crystallographically to represent exact small multiples of a crystal lattice spacing have, time and again, been found interferometrically to be indeed as expected. Thus there is abundant *indirect* evidence of the reliability of contour.

However, it was undoubtedly desirable to examine this point with greater precision. Therefore, with the co-operation of Dr. V. G. Bhide experiments have been conducted in my laboratory recently aiming at an exact numerical evaluation of the perfection of contour obtainable with both silver and multilayer films. The following represents an account of these observations made in these experiments.*

Fig. 1. Silvering arrangement for measuring thin film thickness.

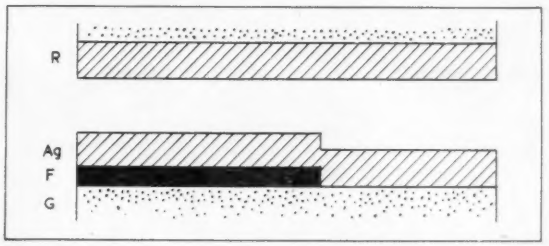

Fig. 2. (× 10) Multiple-beam fringes given by a silver film step of height 520 Å.

The conditions existing in the measurements of

* A survey of the subject has been given by the author in a lecture to the Reunion de Chimie Physique, Paris, in May 1956.

surface microtopography are somewhat similar to those applying to the estimation of the thickness of a thin film. Consider a thin film F on a piece of glass G see Fig. 1. In order to determine the thickness of F a vacuum-evaporated thin but opaque coating of silver Ag, some 1,000 Å thick is deposited on to the system. The resulting step in the silver coating *is supposed* to be exactly equal to the thickness of F. The compound system is then matched against a reference flat R which is coated with a transmitting silver film about 500 Å thick, and by illuminating from above with parallel monochromatic light, fringes are obtained which reveal the step and permit accurate

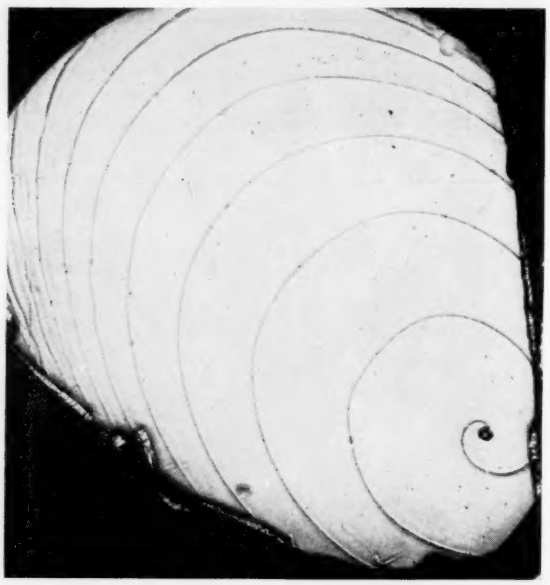

Fig. 3. (×100) A spiral of silicon carbide used as the step.

measurement. A typical example is given in Fig. 2 which shows the fringe displacement obtained by this method for a film thickness of 520 Å.

The validity of the procedure depends clearly on the perfection of contour of the step by the superimposed 1,000 Å film of silver. It will now be described how the degree of perfection of this contour has been tested.

EXPERIMENTAL PROCEDURE

For the purpose of the test it is necessary to have available a *perfectly* defined uniform step with a sharp edge. Further, it must be possible to measure the height of the step accurately before coating. When the measurement is done a silver layer of known thickness is deposited on the step. It will be shown later that it does not matter whether the actual thickness of the silver deposit is strictly in accordance with the estimated thickness, for an *exact* value is not required in this test. The height of the silver-coated step is then re-evaluated. These measurements are repeated successively as the thickness of the silver deposit is increased. As long as the contour is perfect, the measurements will give the same step value, no matter how thick the silver coating which overlies the step. However, should contouring begin to fail at some thickness of the superimposed silver deposit, then the measured step value would indicate the point at which such breakdown sets in.

We have thus before us the formidable problems of securing a *perfect* step, with a natural high reflectivity to enable us to measure the step height interferometrically even before silvering. It would be clearly an extremely difficult thing to prepare such a step by artificial means. Indeed, I cannot think of any satisfactory artificial method. Fortunately nature presents us with precisely the type of a perfect step we are seeking. In connection with a programme of microtopographic studies of a variety of crystals, it has been firmly established in my laboratory that magnificent growth spirals can often be found on crystals of silicon carbide. Such spirals, originating in spiral dislocations, are at times of perfect crystallographic regularity. They consist of a spiral 'ramp' which sweeps round, with vertical 'risers', crystallographically perfect between steps, the steps themselves being normally small integral multiples of the crystal lattice spacing. The crystal has so high a natural reflectivity that quite sharp fringes can be secured even without silvering.

For our experiments we have chosen a spiral which is on a relatively widespread scale and a region of it magnified ×100 is shown in Fig. 3. Spacing between

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the spiral arms is about 0.3 mm. Repeated measurement at many points shows that the spiral step height is 480 ($\pm$ 3) Å, which is a convenient value for these studies.

It is clear from the spiral character that we are dealing with a single spiral. But of course in this picture the optical resolution in extension is small (merely that of a 16-mm microscope objective) hence as a safeguard the step character was examined with an electron microscope (M.E.3) available in this laboratory. Whilst I have little doubt that replication methods are extremely accurate *in extension* I very much doubt the exact validity of replication *in depth.* The great advantage of the electron microscope in my laboratory is that it can be used directly in reflection using grazing illumination without the need of replication. Employing the instrument in that manner one obtains a foreshortened distorted picture with high resolution and high magnification in one direction. This suffices to answer the problem, and Fig. 4 depicts the spiral edge at a magnification of $\times$ 8,000 showing it to be single as expected. A replica (magnification $\times$ 68,000), see Fig. 5, confirms this.

In the experiment silver is deposited on the crystal step and simultaneously on a glass plate alongside this step. The thickness of the silver deposit on the glass plate is evaluated by the method described. Of course, this value of the thickness might be in error due to the very failure of contouring such as we are trying to determine, but an error of this kind can be neglected as it represents only a second order effect and merely causes the axis of the curve in Fig. 7 to shift. As will be shown later, this does not interfere with the substance of our conclusions.

The silvered step height on the crystal is now measured by multiple-beam interferometry. Fig. 6 is a typical example of a fringe pattern and it will be conceded that the fringe definition is so exceptionally good that the step heights can certainly be determined to within one or two Ångstroem units.

In multiple-beam interferometry the silver thicknesses used range from some 500 to 1,000 Å, hence our experiments were begun with a thickness of silver of 250 Å and this was increased successively until a thickness of no less than 26,700 Å was reached. The

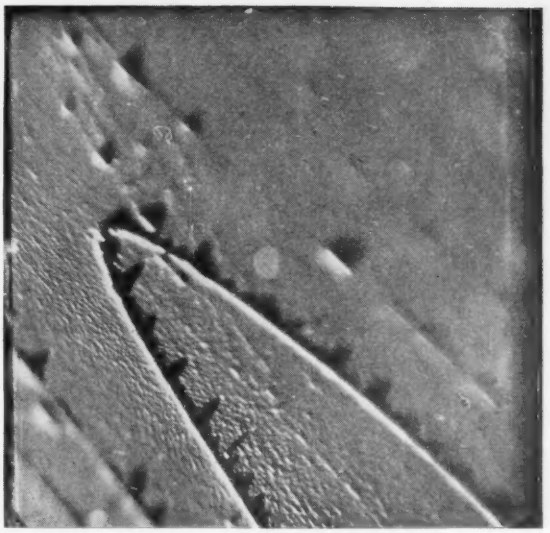


Fig. 4. ($\times$ 8000) Electron microscope picture by direct reflection of silicon carbide spiral. Single step.

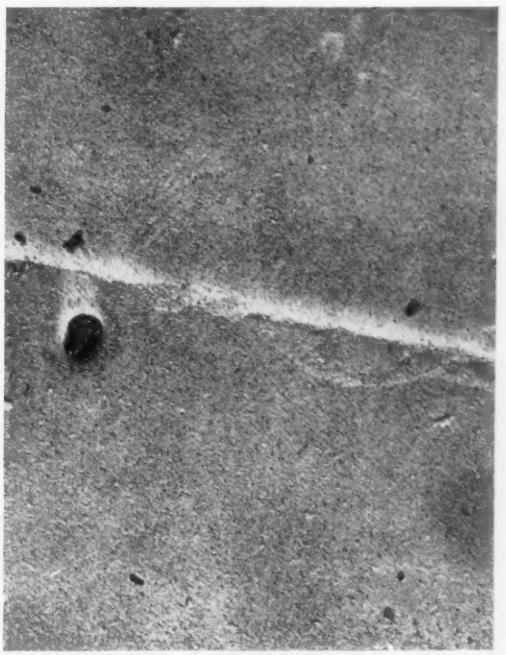
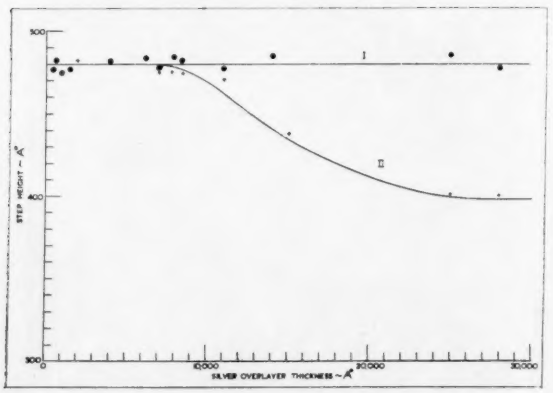


Fig. 5. ($\times$ 68,000) Electron microscope replica of step edge.

Fig. 6. (× 150) Multiple-beam fringes over spiral step.

step height on the crystal was measured at twelve stages within this range of silver thickness.

Two series of step measurements were carried out. In one series the measurement was taken close to the 'riser', *i.e.* at the step edge, and in the other series at a point, 0.075 mm. away from the 'riser'. The latter dimension represents a quarter of the distance between step risers. A small difference was found between the values of the two series of measurements. This will be discussed in detail later.



PERFORMANCE OF SILVER DEPOSITS

The measured values of the step height at different silver thicknesses are given in Fig. 7. The graph shows two curves relating measured step height with silver film thickness.

Curve I corresponds to a measurement made at a point on the 'horizontal' tread which is 1/15th mm. away from the riser. The accuracy of the measurements is very high: The measured step remains consistently at 480 Å for film thicknesses of 250 Å up to 26,700 Å, the error nowhere exceeding 3 Å. In other words contouring is astonishingly perfect. This is remarkable considering the conditions demonstrated in the diagram Fig. 8 which shows the ratio of film thickness to actual step height measured for the thickest film. The silver deposit has a thickness 55 times that of the height of the step, yet exact contouring still persists. This makes it clear why second-order errors in estimating actual silver film thickness can be entirely disregarded. Even major hypothetical errors in this estimation would have little effect on the conclusions reached.

Curve II gives the step height measured at a point as near as possible to the step itself, *i.e.* across the riser. The curve shows that up to a thickness of the deposit of 6,000 Å the measured value of the step still remains precisely 480 Å. At greater thicknesses the measured value of the step progressively decreases by amounts reaching a maximum of 97 Å at an overlayer thickness of nearly 27,000 Å.

Since at a distance of merely 1/15th mm. from the riser the step has its correct value, it is clear that the error is gradually reduced as one proceeds from the edge of the step along the tread. A typical plotting of the changes of the magnitude of the error is given in Fig. 9 which depicts the situation in the case of a silver deposit of 27,000 Å. The magnitude of the error amounting to 97 Å at the edge proper is reduced to zero at a distance from the edge not larger than 15 μ

Fig. 7. Measured step heights for different silver film thicknesses. I at 1/15th mm from riser edge, II very near riser edge.

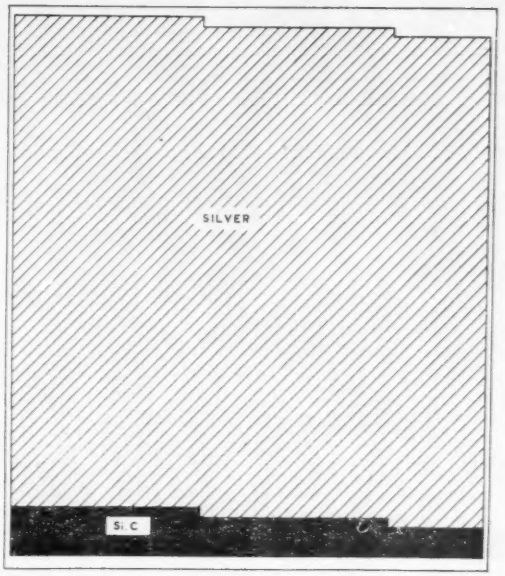


Fig. 8. Scale diagram showing ratio of silver thickness to step height for 26,700 Å silver film.

and from this point onwards the step is correctly contoured.

This signifies that there is a pile-up towards the riser wall.

However, due note should be taken of the fact that below 6,000 Å even this pile-up effect cannot be detected. Normally in multiple-beam interferometry the thickness of the silver film is a mere 500 Å. It never exceeds 1,000 Å. It is clear, therefore, that contouring of microstructure is essentially perfect even very close to the edge.

This result permits two conclusions: (1) A vacuum-deposited silver film of a thickness of less than 1,000 Å certainly gives correct height contouring. (2) In extension, also, the contour is so nearly perfect that one can employ with confidence maximum optical microscope resolution and magnification in extension. Indeed any defects existing in highly magnified fringe patterns say × 2,000, are likely to be due only to known and familiar optical troubles associated with high magnification multiple-beam fringes, and in no way is failure in silver contour the cause of such defects.

The outcome of these experiments completely verify by means of exact measurement the earlier assumptions as to precision contouring based on strong but indirect evidence.

The pile-up on the lower step may possibly be associated with thermal surface mobility and drift towards the vertical wall.

PERFORMANCE OF MULTILAYER DEPOSITS

It has been established before in this laboratory that, in applying multiple-beam interferometry to thin film measurement and topographical studies on opaque bodies, both of which call for the use of *reflection* fringes as distinct from transmission fringes, considerable advantages acrrue from the use of high reflecting zinc sulphide-cryolite multilayers. Such multilayers have a very low absorption and I have demonstrated long ago that the visibility of reflection fringes is determined primarily by the absorption coefficient of the reflector on the matching flat. It is

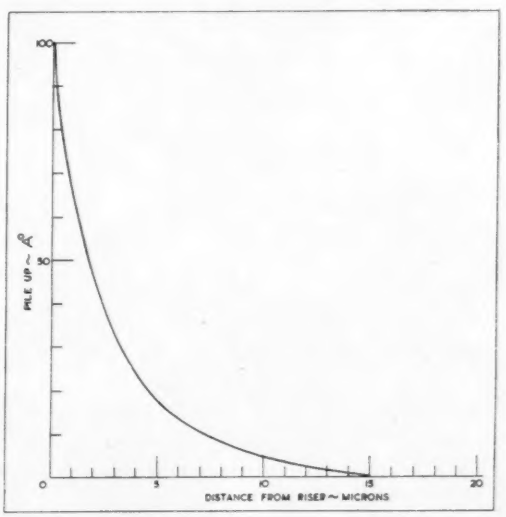


Fig. 9. Variation of pile-up with distance from riser edge for 26,700 Å silver film.

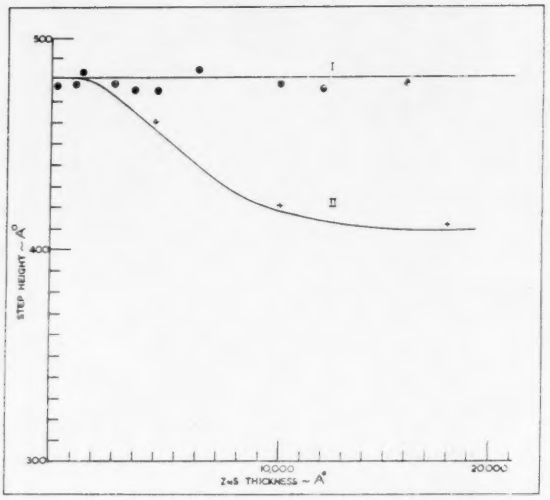
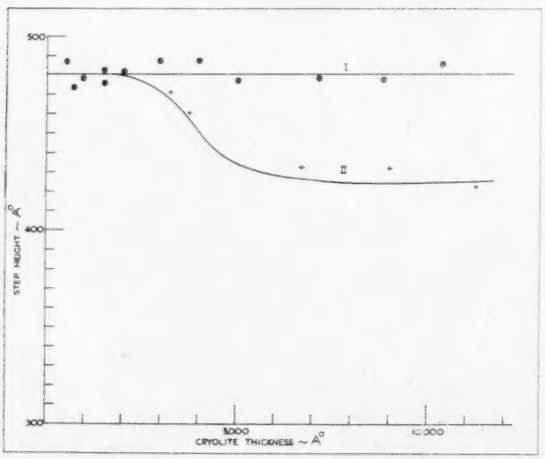


Fig. 10. Measured step heights for different zinc-sulphide film thicknesses. I at 1/15th mm from riser edge, II very near riser edge.

important to note that this restriction on absorption applies only to one of the two mirrors, *i.e.* to the one nearer to the source. Thus high visibility can be obtained without the necessity of coating the surface under study with a multilayer. It is sufficient to coat a surface, say a crystal, with a silver film and the matching (upper) flat with a multilayer to give good visibility.

In this connection it appears worth while considering if fringe definition can be improved by using *two* multilayers. In fact there is a gain in fringe sharpness.



One usually works with green mercury light in this type of experiment and it is difficult to obtain satisfactory reflectivity in the green which is much higher than 94% in the case of silver films, but no difficulty is experienced in obtaining 97% in the case of multilayers. The optics of multiple-beam fringes is such that, at these high reflectivities even a very small increase produces a considerable difference in definition. Two differing surfaces R_1 and R_2 can be treated as equal to a symmetrical pair of reflectivity $R=\sqrt{R_1R_2}$. The fringe half-width is determined by the coefficient of finesse $F=4R/(1-R)^2$ and is approximately $0.63/F^{\frac{1}{2}}$. Two silver layers, a silver-multilayer combination and two nine-layer multilayers can be considered to have reflectivities of 94%, 95.5% and 97% respectively leading to fringe half-widths of about 1/46th, 1/63rd and 1/98th of an order separation.

In other words, the use of one multilayer against one silver film gives in fact a 50% increase in resolving power compared with two silver films, and the use of two multilayers results in another 50% increase in resolution. A total increase in resolution by a factor of two associated with the use of multilayers only is of course, sufficiently important to justify an examination of the contouring efficiency of multilayers.

Multilayers are built up of alternate quarter-wave layers of cryolite and zinc sulphide. The refractive indices of the materials are such that in the green the metrical thickness of a quarter-wave cryolite layer is about 1000 Å and that of a zinc sulphide layer about 600 Å. A film of nine layers therefore has a total thickness of the order of 7,000 Å. This is a formidable thickness compared with a single layer silver film of say 700 Å thickness with a reflectivity of 94%. The question arises whether the gain in reflectivity might not be offset by failure of contouring in view of both the thickness and complexity of multilayers.

Fig. 11. Measured step heights for different cryolite film thicknesses. I at 1/15th mm from riser edge, II very near riser edge.

The contouring efficiency of both zinc sulphide films and of cryolite films have therefore been separately examined using the same silicon carbide spiral as before. The technique had to be modified slightly since neither dielectric has inherently high reflectivity by itself.

The dielectric is deposited on the crystal and on a monitor flat positioned alongside for thickness evaluation. The dielectric is then covered with a top coat of silver about 700 Å thick to give high reflectivity. As mentioned before, no error which may affect the results, is introduced by this procedure. The observed step height then gives a measure of the efficiency of contour. As before, measurements were made at a distance 1/15th mm. from the riser and at the riser itself. The results obtained for zinc sulphide are shown in Fig. 10. Curve I represents the measurements at a point 1/15th mm. from the riser and Curve II the measurements as near as possible to the step edge itself.

The shape of the curves is similar to those obtained for silver deposits but there are some striking differences in numerical values. Measurements could only be carried out on zinc sulphide deposits of a thickness not exceeding about 16,000 Å. Deposits of greater thickness showed a tendency to flocculate. Away from the riser the step height of 480 Å was maintained throughout, although scatter in the measurements was distinctly higher than in the case of silver and a total error range of $\pm$ 5 Å was found. Near the edge itself errors set in at a thickness of about 3,000 Å. At 16,000 Å the error (a reduction in step height) was about 70 Å which is a similar value to that determined on silver deposits of comparable thickness (appr. 60 Å).

Corresponding data for cryolite is shown in Fig. 11. Here again flocculation sets in at thicknesses exceeding 11,000 Å. Scatter is still worse than in the case of zinc sulphide being close on $\pm$ 10 Å.

Errors appeared at some 2,000 Å thickness and quickly reached a maximum of 60 Å at an overall thickness of only 5,000 Å.

It is self-evident from the last two figures that edge effects are more serious in the case of dielectrics than in the case of silver, yet provided the measurements taken in regions nearer than 1/15th mm. of an edge are excluded, reproducibility is fairly reasonable.

Recalling that the total cryolite thickness (split into four layers) is 4,000 Å and the total zinc sulphide thickness 3,000 Å, it is evident that multilayers might lead to grossly inaccurate results of the measurements as long as objects with small detail in extension are studied. This danger is particularly great where elements of a size of the order of about one tenth of a millimetre are concerned. For larger objects such as the evaluation of relatively extended thin films, provided edge effects are ignored, multilayer contouring might well be adequate.

CONCLUDING REMARKS

In general the indication of these experiments seems to be that it is much safer to coat the object with silver and match this against a flat coated with a multilayer. It is better to risk a loss in ultimate sensitivity for the sake of reliability in contour.

Incidentally, the observations recorded in this article seem to offer an explanation why a glass flat covered with a multilayer usually appears to be more smooth than the same flat covered with silver. Obviously, the pile-up mentioned above occurs in the micro-scratches and micro-ruts of the glass flat as well and its effect operates as a smoothing-out mechanism, reducing inequalities.

Vacuum, Vol. IV No. 4 *October, 1954*

PERFORMANCE CHARACTERISTICS OF BARIUM GETTERS

at elevated working temperatures of the valves

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Summary

THE PERFORMANCE CHARACTERISTICS of pure barium getter deposits (bright mirrors only) evaporated from barium aluminium alloys in the temperature range of 100–400°C have been investigated and the results are compared with measurements taken at 20°C and reported in the previous issue. A rise in the operational temperature is shown to have a considerable effect on gas absorption. In the higher temperature regions the getter absorbs gas in quantities closely approaching those required for the formation of the respective chemical compounds. Elevated temperatures appear to cause the molecules of gases, forming an impermeable layer on the getter and thus poisoning it at room temperature, to diffuse into the body of the barium deposit removing such barrier layers entirely. Regeneration of exhausted mirror deposits by means of a heat treatment has been studied. The behaviour of barium mirrors evaporated on glass surfaces heated at 200°C has been examined and some comparative studies have been made between the absorption capacity of freshly deposited mirrors after a heat treatment and prior to such a treatment. The article concludes with observations on gas evolution from getters.

Sommaire

LES CARACTÉRISTIQUES de performance des dépôts getter de baryum pur, (dépôts brillants seulement), évaporés à partir d'un alliage d'aluminium et de baryum à des températures de 100 à 400°C ont été étudiées, et les résultats sont comparés avec ceux obtenus à 20°C, mentionnés dans le journal précédent. On démontre qu'une augmentation de la température d'opération a un effet considérable sur l'absorption des gaz. Dans la région des hautes températures, le getter absorbe les gaz en quantités presque semblables à celles nécessaires pour la formation des composés chimiques respectifs. Les températures élevées semblent agir, en sorte que, les molécules de gaz formant une couche imperméable sur le getter, et de ce fait l'empoisonnant à la température ambiante, se diffusent dans le getter et ainsi suppriment de telles barrières. On a étudié la régénération de dépôts mirroirs. Le comportement de mirroirs de baryum évaporés sur des surfaces de verre chauffées à 200°C a été examiné, et quelque études comparatives ont été faites entre la capacité d'absorption de mirroirs fraichement déposés, avant et après le traitement thermique. Cet article se termine avec quelques observations sur l'évolution de gaz provenant des getters.

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INTRODUCTION

THE WORK REPORTED in this article forms an extension of the investigation[1] published in the last issue of 'Vacuum'. As before, the subject is the absorption capacity of barium mirrors evaporated from 50 : 50 barium-aluminium alloys. But, while we concentrated initially on getter performance at room temperature, we are now concerned with getter behaviour at higher temperatures, up to 400°C.

No change has been made to the experimental method employed, and the equipment used in the present investigation is identical to that shown in Fig. 1 of the previous article (reproduced on the opposite page) except for a thermostatically controlled heater which has been fitted to the getter chamber in order to raise the temperature of the barium deposits in-

* MS. received April, 1957.

vestigated, to the desired values.

The merits of the revised capillary method have been discussed at great length in the first paper. Thus it is only necessary to state that the experiments referred to in the present report have been carried out invariably at constant pressure in the getter chamber, *i.e.* keeping P_g=constant. As mentioned then, the method yields capacity values of direct practical significance but has never before been used for a methodic exploration of getter capacity characteristics. We believe therefore that some of the data submitted below is of a truly novel character.

PRACTICAL SIGNIFICANCE OF HIGH TEMPERATURE PERFORMANCE

In the previous investigation[1], we dealt with the conditions as they present themselves when the mirror deposit is at a temperature of 20°C. Few electronic valves work at temperatures as low as that but nevertheless these conditions are of significance in practice. For instance, in the normal type of cathode-ray tubes the getter deposit is substantially at room temperature because the mirror is comparatively far away from the cathode and well protected against heat radiation from other electrodes. To be precise, there may be a slight but insignificant rise in temperature, of, say, 10 or 20°C above room temperature due to heat dissipation from the cathode and heat developed in the cabinet.

Television tubes are a special case. Here, absorption capacity is governed by additional factors such as the type of surface, aluminium or graphite, on which the getter is deposited, and the geometrical arrangement available for getter evaporation. The influence of these factors is outside the scope of the present investigation and therefore the information given in this article does not apply to conditions in t.v. tubes.

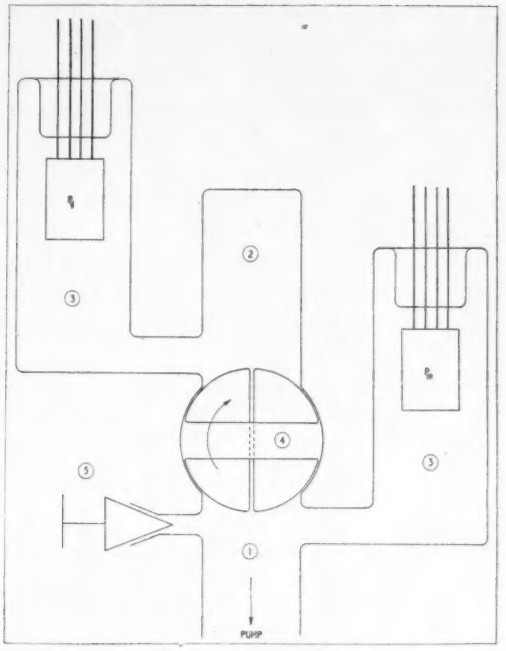
The essential purpose of the present investigation was to explore operational conditions in receiver and small transmitter valves. Modern design trends in this field aim at compactness and reduced dimensions with the result that working temperatures are rising. The following measures have been taken, facilitating higher working temperatures:

(1) Modern valves have an all-glass envelope and the plastic base which limits its working temperature has been eliminated.

(2) All metal components are made from high-purity grade metals or from metals which can be degassed without difficulty.

(3) The materials employed for the anodes are capable of high heat dissipation.

(4) Magnesium getters, which are liable to re-evaporation after deposition, have been dispensed with and more efficient getters have been introduced.



(Fig. 1. reprinted from page 288). Diagram of the experimental equipment. *Legend:* (1) Manifold, (2) getter chamber, (3) ionisation gauges, (4) valve carrying capillary, (5) needle valve. The valve (item 4) facilitates fast evacuation of the equipment.

The working temperature in some types of valves now reaches almost 200°C. Certain areas of the barium mirror in such valves may reach even higher temperatures due to the heat developed by the cathode. It is important, therefore, to obtain information on the behaviour of barium mirrors at any level in the temperature range of 20–400°C, as it may have

an appreciable effect on valve performance.

It was shown in the previous paper that certain gases are absorbed by the getters in considerable quantities whereas other gases produce early saturation of the getter and absorption ceases abruptly. If one considers the working conditions in modern valves and takes into account the amount of gas evolved during their operation, the total absorption capacities at 20°C, shown in Table 1, appear to be rather too low to justify expectations of a reasonably long service life. As will be seen below, the present experiments furnished evidence of the fact that the absorption capacity of barium deposits in such valves is appreciably higher than previously assessed, and that this improvement has its sole and only cause in the increased working temperatures.

Table I. Total Absorption Capacity of Bright Barium Mirrors

Gases	*Quantity absorbed at*				
	20°C	100°C	200°C	300°C	400°C
Oxygen ..	50 1μ/mg.			57 1μ/mg.	
Moisture ..	35 1μ/mg.			72 1μ/mg.	
Hydrogen ..	4.48 1μ/cm.^{2}	5 1μ/cm.^{2}	5.5 1μ/cm.^{2}	9 1μ/cm.^{2}(90 1μ/mg).	10 1μ/cm.^{2}(100 1μ/mg).
Carbon Dioxide	0.60 1μ/cm.^{2}	2 1μ/cm.^{2}	3.3 1μ/cm.^{2}	5.8 1μ/cm.^{2}(58 1μ/mg).	6.6 1μ/cm.^{2}(66 1μ/mg).
Carbon Monoxide	0.72 1μ/cm.^{2}	4 1μ/cm.^{2}	4.4 1μ/cm.^{2}	9 1μ/cm.^{2}(90 1μ/mg).	10 1μ/cm.^{2}(100 1μ/mg).
Nitrogen ..	0.33 1μ/cm.^{2}	0.48 1μ/cm.^{2}	3.4 1μ/cm.^{2}	5 1μ/cm.^{2}(50 1μ/mg).	5.1 1μ/cm.^{2}(51 1μ/mg).
Dry Air ..	0.64 1μ/cm.^{2}	1.60 1μ/cm.^{2}	4.6 1μ/cm.^{2}	5.6 1μ/cm.^{2}(56 1μ/mg).	5.6 1μ/cm.^{2}(56 1μ/mg).

EXPERIMENTAL RESULTS AND DISCUSSION

During the planning of the experiments great care was taken to simulate conditions in practice as closely as possible. After baking the system, the outgassing valve was closed and the capillary, forming the only communication between the getter chamber and the manifold, was inserted. A quantity of 1 mg. of getter material was evaporated in each run, giving a deposit of 10 sq. cm., *i.e.* a weight-to-area ratio of 0.1 mg. per sq. cm. The getter was fired at a pressure of 1×10^{-4} mm. Hg. The evaporation took 7 seconds. After firing the getter, the heater was switched on to raise the temperature of the barium mirror to the desired level. The chosen temperature could be maintained with an accuracy of ± 2°C by means of a precision thermometer controlling the thermostat. When temperature equilibrium had been established, the gas to be tested was admitted to the system *via* the needle valve and the absorption measurements were started.

Detailed results of the experiments are given in Table 1 and in graphs, Figs. 1a–g. The following comments are made to assist in their interpretation.

Oxygen

Table 1 shows the total absorption capacity at 20°C and 300°C. Fig. 1a shows the values of the instantaneous absorption capacity.

The theoretical amount of oxygen required to effect complete chemical conversion of 1 mg. of barium into barium oxide is 68 1μ. Compare this figure with the two values shown in Table 1 for 20°C and 300°C. The graph clearly demonstrates that a rise in temperature accelerates absorption. These results agree with those obtained by Bloomer during recent experiments[2,3] employing the capillary method but keeping the pressure in the manifold (p_m) constant. Our experiments also confirm Bloomer's observations that barium mirrors evaporated in a very high vacuum will start absorption activities only after deterioration centres have been produced. This phenomenon is particularly marked in the case of oxygen, carbon monoxide and carbon dioxide, but it can also be observed with other gases, if the conventional method

of p_m=constant and low-conductance capillaries are used.

The formation of a barium oxide layer on the mirror surface is indicated by a milky white appearance of the mirror.

Moisture

Table 1 shows the total absorption capacity at 20°C and 300°C. Fig. 1b shows the values of the instantaneous absorption capacity.

Both sets of values show that as the temperature rises the quantities absorbed increase and approach the theoretical amount required for total conversion into $Ba(OH)_2$ and BaH_2 (approximately 130 $l\mu$/mg.). The instantaneous absorption capacity is essentially constant over a long period.

At 300°C the appearance of the mirror changes into a semi-transparent cloudy reddish brown.

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Hydrogen

Table 1 shows the total absorption capacities at 20°C, 100°C, 200°C, 300°C and 400°C. Fig. 1c shows the values of the instantaneous absorption capacity.

The theoretical amount required to effect complete chemical conversion into BaH_2 is 135 $l\mu$/mg. It will be noted that the total capacity of the getter for hydrogen at 20°C is already between one third and one quarter of the theoretical amount required and there is little change in the getter behaviour at temperature levels of 100 and 200°C. In order to increase the absorption capacity it is necessary to raise the temperature to 300 and 400°C, at which levels measured and theoretical values are very close to each other.

At high temperatures the mirror assumes a semi-transparent brown appearance.

Carbon Dioxide

Table 1 shows the total absorption capacities at 20°C, 100°C, 200°C, 300°C and 400°C. Fig. 1d gives the values of the instantaneous absorption capacity.

In this case it is not possible to refer to any amounts required for chemical conversion as it is not known what compound would result from the reaction between carbon dioxide and barium. It can be seen from Table I, and the graphs that the quantities taken up by the getter at 300°C are nine to ten times the amount taken up at 20°C. At 400°C absorption is accelerated appreciably.

It will be remembered that measurements at room temperature indicated an early cessation of the absorption activity of the getter for both gases, carbon dioxide and carbon monoxide. This getter poisoning effect was traced back to the formation of an impervious barrier layer on the surface of the getter deposit, preventing the body of the barium deposit from sharing the absorption activities. The outcome of the present in experiments shows that there is a substantial change in the absorption mechanism when the temperature is raised. High temperatures appear to facilitate diffusion of the gas into the body of the deposit thus facilitating re-activation of the top surface of the mirror. The behaviour of the barium getter in this instance shows close similarity with the general absorption mechanism of other metals at high temperatures, for instance, zirconium and titanium. It seems that raising the temperature affords the one and only means of preventing the formation of the barrier layers mentioned.

At 200°C, when the absorption activities come to an end, the metallic appearance of the getter deposit begins to deteriorate. At 300°C the deposit assumes a semi-transparent brownish appearance and at 400°C the metallic appearance has disappeared entirely.

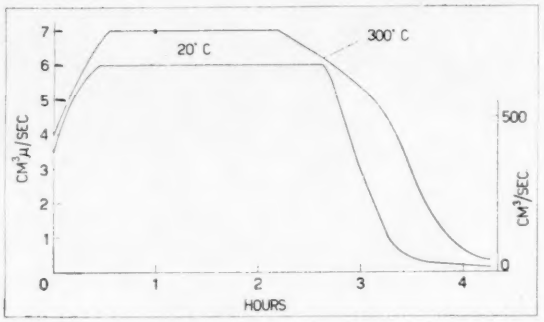
Carbon Monoxide

Table I gives the total absorption capacities at 20°C, 100°C, 200°C, 300°C and 400°C. Fig. 1e gives the values of the instantaneous absorption capacity.

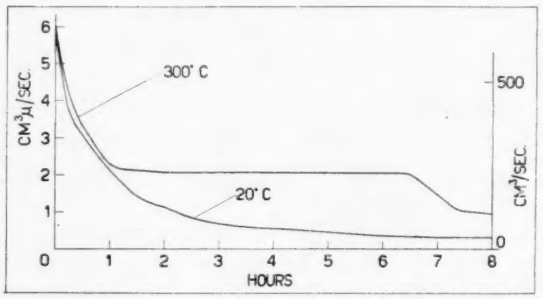
At 100°C the amount absorbed by the barium mirror is six times that absorbed at 20°C, and at 300°C the amount is twelve times that absorbed at 20°C.

The poisonous properties of carbon monoxide at room temperature, originally indicated by Wagener[5], have been confirmed in our experiments described in the previous paper. In a recent investigation Stoll[6] established that carbon monoxide is the gas most amply evolved during the service life of the thermionic tube. The problem of removing carbon monoxide

Values of the Instantaneous Absorption Capacity
Determined for Various Gases at Elevated Temperatures Keeping p_g = constant



(*a*)



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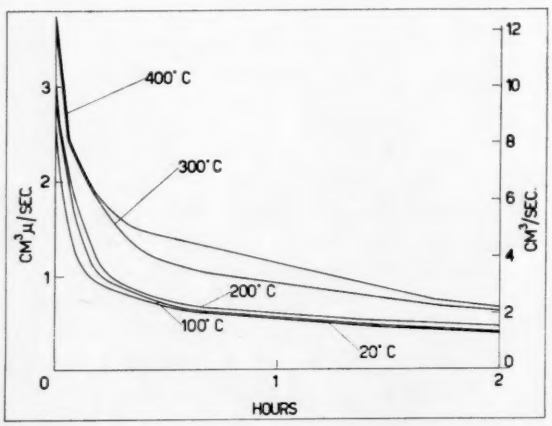

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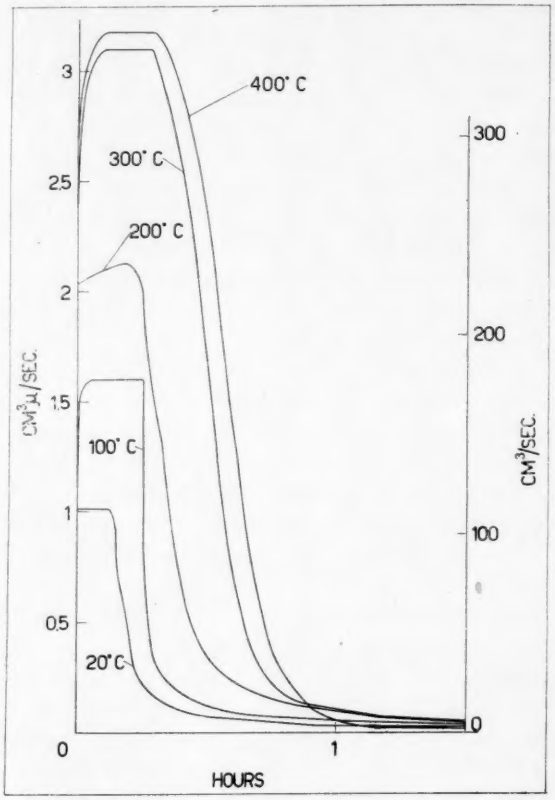

(*d*)

Captions to Figure 1

(*a*) Oxygen. $p_g = 9 \times 10^{-6}$ mm.Hg. Weight of barium:—1.5 mg.

(*b*) Moisture. $p_g = 9 \times 10^{-6}$ mm.Hg. Weight of barium:—1.4 mg.

(*c*) Hydrogen. $p_g = 9 \times 10^{-6}$ mm.Hg. Mirror area:—1 cm.² Weight of barium:—0.1 mg.

(*d*) Carbon Dioxide. $p_g = 9 \times 10^{-6}$ mm.Hg. Mirror area:—1 cm.² Weight of barium:—0.1 mg.

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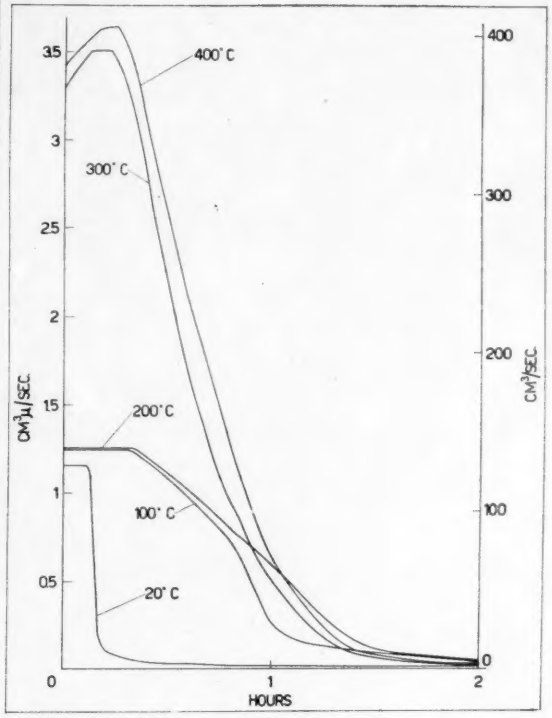
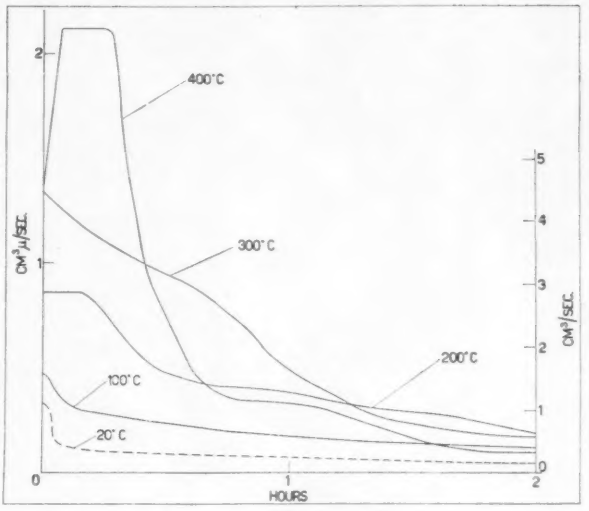
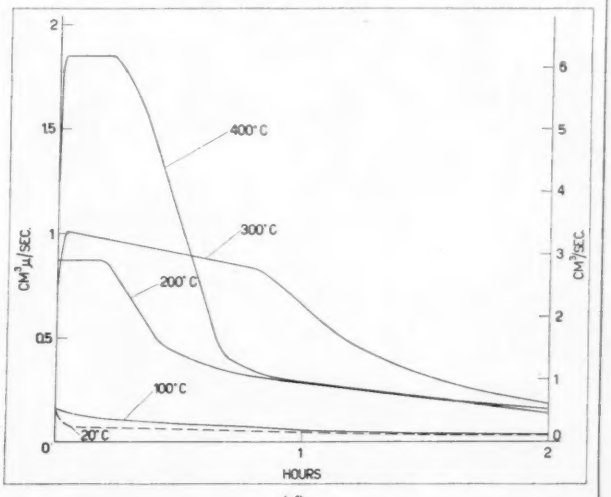
Values of the Instantaneous Absorption Capacity
Determined for Various Gases at Elevated Temperatures Keeping p_g = constant



(*e*)



(*g*)



(*f*)

Captions to Figure 1

(*e*) Carbon Monoxide. $p_g = 9 \times 10^{-6}$ mm.Hg. Mirror area:—1 cm.² Weight of barium:—0.1 mg.

(*f*) Nitrogen. $p_g = 9 \times 10^{-6}$ mm.Hg. Mirror area:—1 cm.² Weight of barium:—0.1 mg.

(*g*) Dry Air. $p_g = 9 \times 10^{-6}$ mm.Hg. Mirror area:—1 cm.² Weight of barium:—0.1 mg.

Values of the Absorption Capacity

of Mirrors Exhausted at Room Temperature, when Subjected to Elevated Temperatures. (Barium Mirror = 1 cm.²)

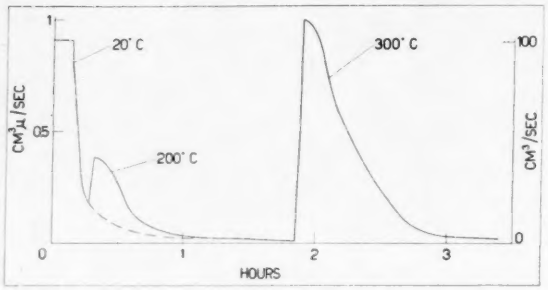
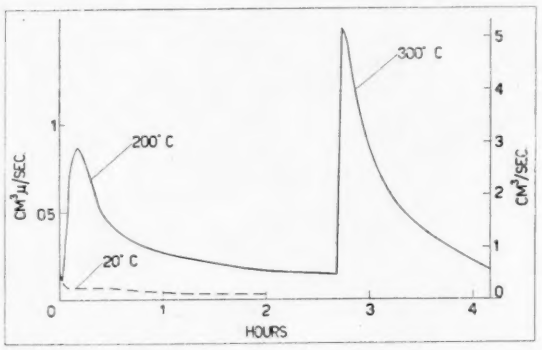


Fig. 2 (*a*) above. *Fig.* 2 (*b*) below.



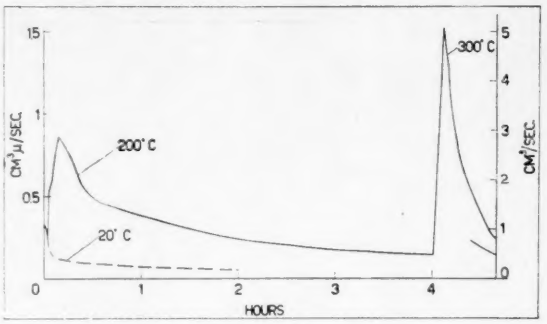


Fig. 2 (*c*)

Captions to Figure 2

(*a*) Carbon Dioxide. $p_g = 9 \times 10^{-6}$ mm.Hg.

(*b*) Nitrogen. $p_g = 3 \times 10^{-4}$ mm.Hg.

(*c*) Dry Air. $p_g = 3 \times 10^{-4}$ mm.Hg.

Values of the Absorption Capacity

of Mirrors, Regenerated by Heat Treatment, at Room Temperature
(Barium Mirror = 1 cm.²)

Fig. 3 (*a*) Dry Air. $p_g = 3 \times 10^{-4}$ mm.Hg. Heating to 200° C.

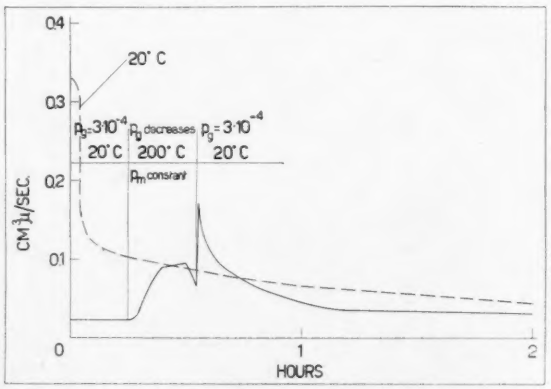
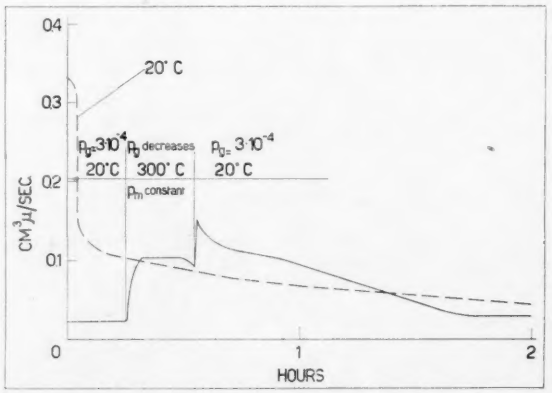


Fig. 3 (*b*) Dry Air. $p_g = 3 \times 10^{-4}$ mm.Hg. Heating to 300° C.



is thus a major one. Morrison and Zetterstrom[4] investigated in 1955 the absorption capacity of barium for carbon monoxide employing the capillary method and keeping the pressure in the manifold (p_m) constant. These workers came to the conclusion that a change of temperature has no effect on the amounts of carbon monoxide absorbed by barium deposits. Obviously our observations do not agree with these conclusions.

At 300°C the appearance of the mirror begins to change in a manner similar to the changes described for carbon dioxide.

Nitrogen

Table 1 shows the total absorption capacities for nitrogen at 20°C, 100°C, 200°C, 300°C and 400°C. Fig. 1f shows the values of the instantaneous absorption capacity.

The theoretical amount required for the conversion of 1 mg. of barium into Ba_3N_2 is 41.3 1μ/mg. This figure however is lower than that required to form $Ba(N_3)_2$. It can be seen that the total amount absorbed at 400° is higher than that theoretically required to form Ba_3N_2. It is interesting to note that at 100°C the absorption capacity for nitrogen is only slightly raised. Larger rises can only be effected at higher temperatures.

Air

Table 1 gives the total absorption capacities at 20°C, 100°C, 200°C, 300°C and 400°C. Fig. 1g gives the values of the instantaneous absorption capacity.

Due to the presence of oxygen in air besides nitrogen, the absorption characteristics are slightly different from those of nitrogen, in particular in the region of lower temperatures where the absorption is somewhat better.

THE EXHAUSTED GETTER

Many investigations into the failure of thermionic valves have established a rise of the pressure of the residual gas atmosphere in the valve as the primary cause. A gradual rise of the residual gas pressure, besides its potential effects on the characteristics of the valve, constitutes a particular danger with respect to the cathode because the emission will be reduced due to poisoning by free barium and eventually the cathode will be destroyed by ion bombardment. In many instances cathode poisoning is reversible and remedial action in the form of raising the cathode temperature is successful, provided the sources of poisoning can be removed. No doubt in a great number of cases impaired activity of the getter accounts for pressure rises or fluctuations occurring in the valve. Therefore it is worthwhile investigating whether, in such cases, a tolerable degree of vacuum can be restored in the tube by devising a treatment of the barium deposits, regenerating, at least partially, their gettering action.

Below we give observations made during a series of experiments aimed at exploring these conditions.

Method of Regeneration

Barium deposits which seem to be completely exhausted and no longer capable of absorbing gas at ambient temperature were subjected to a heat treatment increasing the temperature stepwise. The experiments were extended to conditions where the getter is exposed to carbon dioxide, see Fig. 2a, to nitrogen, see Fig. 2b, and to dry air, see Fig. 2c. The values recorded indicate that absorption is resumed when the temperature is raised. These observations suggest a very simple method of improving vacuum conditions in a valve which has filled up with residual gases in the course of its use. Satisfactory regeneration of the barium deposit can be obtained by placing the valve in a furnace and heating it at 300°C for about one hour. An alternative procedure would be to play a gas flame of about 300°C for one hour on the area of the valve envelope which supports the mirror deposit.

Performance of Regenerated Getters

Having established that heat treatment of the valve successfully restores the vacuum conditions in the valve it is important to know whether a barium deposit, regenerated in this manner, is capable of absorbing gases at normal operational temperatures

Values of the Absorption Capacity
of Mirrors, Deposited on Glass Heated at 200°C (Barium Mirror = 1cm.²)

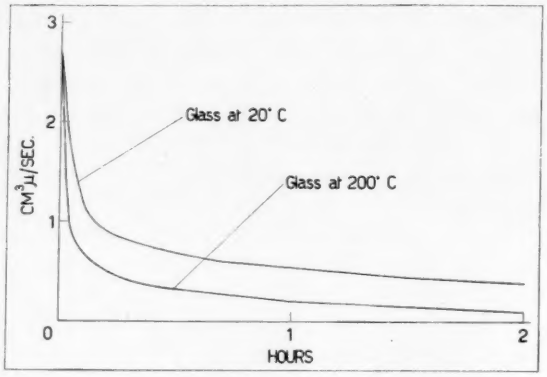


Fig. 4 (*a*)

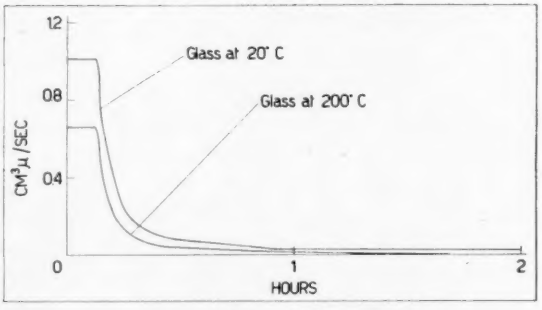


Fig. 4 (*b*)

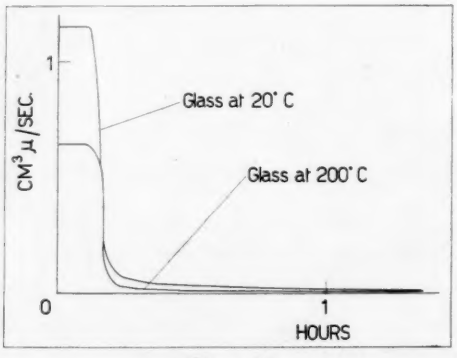


Fig. 4 (*c*)

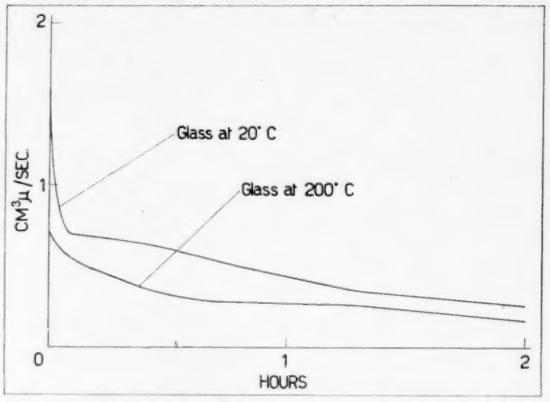


Fig. 4 (*d*)

Captions to Figure 4

(*a*) Hydrogen. $p_g = 3 \times 10^{-4}$ mm.Hg.

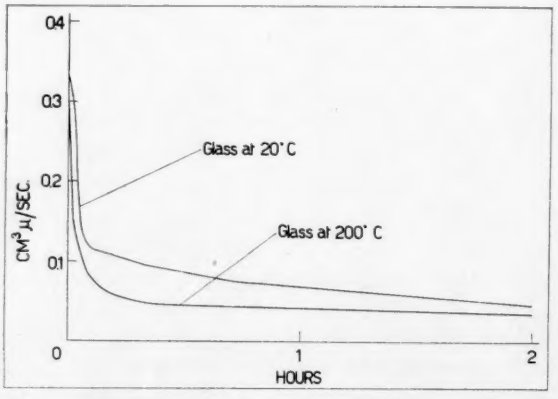
(*b*) Carbon Dioxide. $p_g = 9 \times 10^{-6}$ mm.Hg.

(*c*) Carbon Monoxide. $p_g = 9 \times 10^{-6}$ mm.Hg.

(*d*) Nitrogen. $p_g = 3 \times 10^{-4}$ mm.Hg.

(*e*) Air. $p_g = 3 \times 10^{-4}$ mm.Hg.

Fig. 4 (*e*)



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of the valve. In order to obtain as accurate information as possible on this point we chose the most unfavourable working temperature, *i.e.* room temperature, as in the case of cathode-ray tubes, for instance. Tests with valves heat-treated for a quarter of an hour at 300°C showed that the absorption capacity of the mirror was considerably better than prior to the heat treatment but somewhat below that of a freshly deposited mirror. This is clearly demonstrated in Figs. 3a and b by comparing the results of the tests with the absorption capacity values for fresh barium deposits at 20°C.

The outcome of these experiments permits the conclusion that it is possible to regenerate exhausted barium deposits in cathode-ray tubes, but in practice it appears advisable not to confine remedial action to the heat treatment and to add the further processing step of evaporating some fresh barium in the valve. For reasons explained in detail below, this additional evaporation process should be carried out while the valve is cold.

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STRUCTURE OF THE GETTER DEPOSIT

Effect of Heating the Glass during Deposition

The question has often been raised at what temperature of the glass envelope the getter should be evaporated. It is known in the field of vacuum evaporation technique that deposition on a hot substrate produces a film consisting of large crystals. However, if the substrate is kept cool during deposition, the size of the crystals comprising the film is small. The same applies to the evaporation of getters. There is the additional consideration that a mirror consisting of small crystals can be expected to absorb a larger quantity of gas than a deposit consisting of large crystals. The results of a series of experiments conducted to verify this hypothesis are shown in Figs. 4a-e. The experimental values recorded in the graphs refer to barium deposition on glass at 200°C. The dotted curves in the graphs show the absorption capacity at room temperature. It can be seen that the absorption capacity of the getter deposited on a hot glass envelope is about half that deposited at room temperature.

Performance of Fresh Deposits After Heat Treatment

In order to obtain additional proof that there is an intimate relationship between absorption capacity of the getter and crystal size of the deposit, a further series of experiments was carried out, the results of which are shown in Figs. 5a–b. The absorption capacities recorded in these graphs refer to barium mirrors evaporated at room temperature and heated subsequently to 350°C before they were exposed to a gaseous atmosphere. The reduced absorption capacities confirm that heat treatment at high temperatures causes re-crystallisation in the deposit resulting in large crystals with an attendant reduction in gettering power.

These results serve as a further illustration of the phenomena discussed in the previous section, and might also explain why we have been unable to achieve complete regeneration of an exhausted getter by heat treatment. It is now clear that heat treatment causes diffusion of the absorbed gases into the body of the deposit limiting mirror regeneration to the surface of the deposit. At the same time larger crystals may be formed which would cause the overall absorption capacity of the mirror to be lower.

OBSERVATIONS ON GAS EVOLUTION

Before summing up the significance of the result of the present work we would like to make a few remarks on the topic of gas evolution from getters.

No gas evolution has ever been observed during any of the experiments reported here. This is a firm indication that the mechanism of gettering is a process of chemical conversion and not a physical process as claimed by many authors in the past. At any rate, there would be no danger from evaporation of barium at high temperatures since the barium vapour pressure at 400°C is only 5×10^{-7}mm. Hg and therefore can be neglected.

Operating at temperatures higher than those employed in the present experiments, *i.e.* at 500°C, the author did in fact observe considerable evolution of gas. But in looking for the causes it should not be forgotten that the all-Pyrex experimental equipment

was degassed at temperatures not exceeding 450°C and therefore a large portion of the gas evolved may have originated from the walls of the plant.

Values of the Absorption Capacity of Mirrors, at Room Temperature and Heat Treated Prior to Exposure (Barium Mirror = 1 cm.²).

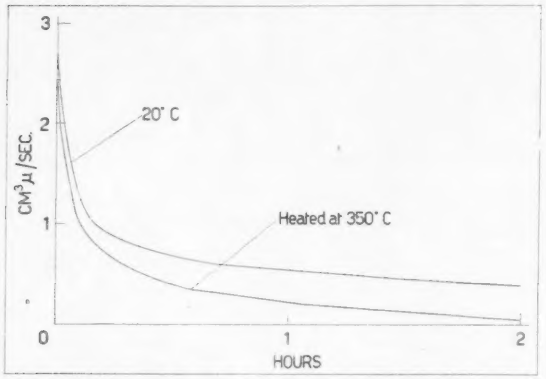
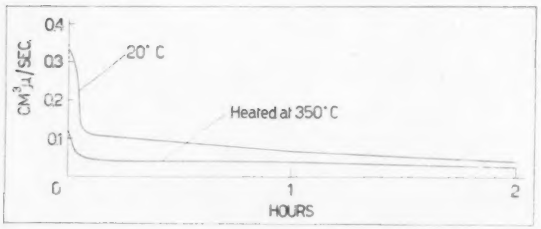


Fig. 5. (*a*) Hydrogen. $p_g = 3 \times 10^{-4}$ mm.Hg.

Fig. 5. (*b*) Air. $p_g = 3 \times 10^{-4}$ mm.Hg.



CONCLUSIONS

The work reported here forms an extension of work described in the previous issue[1] of this journal on the performance characteristics of pure barium deposits obtained by the evaporation of a barium-aluminium alloy.

The behaviour of getter deposits at temperatures ranging from 100–400°C permits the following conclusions:

(*a*) An operational temperature above room temperature has an appreciable effect on the absorption capacity available.

(*b*) Given sufficient time and the appropriate operational temperature, a pure barium deposit will reach complete exhaustion depending only on the individual absorption characteristics with regard to the gases concerned.

(*c*) At increased temperatures the barium mirror will absorb a gas in quantities approaching those required for the formation of a chemical compound between the barium and the respective gas.

(*d*) High operational temperatures permit elimination of the poisoning effect observed with certain gases at room temperature since high operational temperature prevents the formation of barrier layers.

(*e*) A barium mirror deposited on a hot surface shows lower absorption capacity than a mirror deposited on a surface kept at room temperature. This phenomenon appears to be related to the formation of large crystals in the mirror if deposited on a hot surface. Similar conditions have been observed if barium mirrors deposited at room temperature have been heat-treated prior to their exposure to gas.

(*f*) Operational temperatures up to 400°C do not cause any gas evolution from the barium deposits.

Of particular significance to production conditions in valve making are the following points related to the above findings:

(*a*) The use of pure barium mirrors facilitates working temperatures in valves of the order of 200°C and over.

(*b*) It is possible in practice to restore the vacuum within a valve by heat treatment of the barium deposits at temperatures about 200–250°C above the working temperature of the valve for a period, the length of which depends on the quantity of gas to be absorbed.

(*c*) Exhausted barium mirrors can be at least partially regenerated by heating the deposit at a temperature of about 300°C for a certain period.

(*d*) It is preferable to evaporate barium getters

onto cold surfaces rather than hot surfaces.

These results should be of considerable assistance in reducing the number of rejects experienced in valve production.

As in the case of the previous report the author wishes to express his appreciation for the assistance in the experimental work given by Dr. Origlio, a member of this laboratory.

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LIQUID HELIUM IN BRITAIN

ORGANISATION AND PURPOSE OF THE LIQUID HELIUM POOL

Sommaire

LE 'LIQUID HELIUM POOL' est une organisation pour mettre en commun la liquéfaction de l'hélium. Le liquide, transporté dans des vases de Dewar, est distribué par route ou chemin de fer aux laboratories l'utilisant. Il est possible que cette organisation ait grand effet sur le future de la recherche cryogénique en Angleterre. Description est faite de l'organisation et de son financement, ainsi que des techniques et installations employées.

Introduction

Low temperature research is nowadays usually taken to mean research below 20°K. Liquid hydrogen boils at about this temperature under atmospheric pressure; by reducing the pressure, the temperature can be lowered to about 10°K. Liquid helium can cover the range between 4.2°K. and 1°K. The gap between the two ranges can be covered by a variety of techniques.

An organisation—the Liquid Helium Pool—has come into being in the last few years for supplying liquid helium for research purposes. Laboratories making use of this service are saved the trouble of liquefying the gas themselves and can concentrate on the experimental work at low temperatures with the minimum of effort.

In order to explain the full significance of this idea, this article starts with a very brief resumé of the history of low temperature research in Universities and of its gradual utilisation by industrial and other laboratories. Recent articles[1, 2] have dealt with present-day applications of low temperature research and this topic will not be touched on here. The main purpose of the present paper is to describe the organisation of the Liquid Helium Pool which has, up to now, only received brief mention in the literature[3], the technique of handling the liquid helium and the equipment needed to start a laboratory using it.

Historical Background

By 1860, the basic principles of refrigeration were perfectly well understood. The absolute scale of temperature, the Joule-Kelvin effect and the principles of the refrigerator and heat pump were known. Interest was then focused on the problem whether all gases could be liquefied, as the theory of the atomic structure of matter predicted they should. So, one after the other, the 'permanent' gases succumbed to cold. It was in 1877 that Cailletet first produced a mist of oxygen droplets. Progress was impeded by purely practical considerations—the difficulty of obtaining good quality tubing, the poor performance of pumps and compressors and the absence of experience in vacuum technique. At that time there were, of course, no Dewar flasks and it was the ambition of one of the leading scientists to produce a few cubic centimetres of the liquefied gas 'boiling quietly in a test tube'.

About 1891 Dewar invented his flasks and seven years later he liquefied hydrogen. In 1908 Kamerlingh Onnes at Leiden liquefied helium and reached a temperature of about 1.7°K at the first try. At this time academic research was focused on aspects of quantum theory—indeed the discovery that the specific heats of solids decreased rapidly with falling temperature was one of the most important disagreements with classical theory that had come to light.

The industrial development which paralleled these academic achievements was slow at first. In 1890 a Bessemer convertor in Glasgow was fed with oxygen gas; the bottom blew out and the charge disappeared. By 1906 the British Oxygen Company had a plant capable of producing 500 cubic feet of oxygen per hour by rectifying liquid air. It stood beside another which made oxygen by the barium oxide process. Liquid oxygen was used only in mine rescue work and in aircraft and to a small extent in blasting operations; nitrogen was wanted for ammonia production. By 1920, metal Dewars containing charcoal getters in the interspaces were used for transport. Oxyacetylene cutting and welding came into vogue a little later and have dominated industrial liquefaction ever since. The iron- and steel-making industries are now beginning to use large quantities of oxygen. Already they need 20% of the total production and in the next few years will probably become the largest users.

On the academic side, Kamerlingh Onnes' laboratory at Leiden was for 15 years the only one in the world to produce liquid helium. He had a tiny budget to work with, but in spite of this his laboratory produced a steady stream of discoveries. By the 1930s a number of other institutions had begun utilising liquid helium—the first were Toronto, Berlin, Cambridge and Washington. Miniature liquefiers and small-scale techniques were developed, notably at Berlin and Oxford, to defeat financial

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stringencies. But low temperatures remained something of a very specialised craft. In particular, the problems being attacked were restricted in scope. Mostly they were problems in magnetism and the macroscopic manifestations of quantum theory, such as the superconductivity of some metals and the superfluidity of liquid helium.

The Creation of the Pool

After the second world war, there was a marked decrease in the thermometric lag of industrial behind academic research. Partly this was due to the increased sponsoring of pure research by Government and industry, but above all it was due to the development of a commercially manufactured helium liquefier in 1947 due to Collins of the Massachussetts Institute of Technology[1]. The design was evolved partly out of wartime experience with portable oxygen liquefiers and later with an enormous hydrogen liquefier. The machine was marketed by the Arthur D. Little Company, a firm which exploits the inventions of M.I.T. Initially it was estimated that perhaps three machines might be sold, but since that date about 60 have been bought by Universities, industrial research laboratories and Government establishments throughout the world. The cost is in the region of $30,000 each. The machine is delivered complete with all accessories and auxiliary apparatus and, in the words of one eminent physicist, low temperature research has been brought within the reach of anyone who can pay for it. The millions of dollars which the U.S. Government has poured into low temperature research in the past ten years contrast strangely with the attitude that Kamerlingh Onnes met.

At about the same time another invention finally took much of the remaining difficulty away from research with liquid helium. This was the remarkable design of a metal Dewar evolved principally by Wexler and his associates at the Westinghouse Research Laboratories at Pittsburgh[5]. Whereas liquid helium had always been handled with extreme care and had been regarded as such a volatile liquid that, by extreme coddling, it might be kept for a day or two at most, it was now possible to store litres of it for months. Instead of having to be used within a short time of being liquefied it could be made in advance and then used at leisure.

The ten-fold increase in the number of low temperature laboratories throughout the world has had a profound influence on the type of research being carried out. There is a great emphasis on applied research, extending measurements of the most diverse kinds down to lower temperatures. The low temperatures are often regarded as just one research technique out of many which can be used in attacking a given problem in, say, solid state physics or nuclear physics.

The post-war expansion of facilities in this country was very slow compared with that in America. Four new laboratories equipped themselves to make liquid helium and all did so by building their own liquefiers. Though this can be done for a fraction of the cost charged for the manufactured article, the construction of a liquefier demands some skill and occupies a fair amount of time. On the other hand, few public or private research institutions in this country can afford the sterling equivalent of $30,000, let alone the dollars themselves, for a single piece of equipment.

The situation was quite changed when in 1952 the National Physical Laboratory (N.P.L.) was presented with a Collins liquefier under the Marshall Aid Programme. Later it was paid for out of a fund which facilitated payment in sterling. It had been intended to use this for calorimetric measurements of interest to chemical engineers. But it was soon realised that it was possible, in addition, to put it to far greater use—namely for what is now known as the Liquid Helium Pool. The idea was that helium should be regularly liquefied in bulk and then sent by road or rail to any laboratory. Such operations were thought to be feasible if well designed Dewars were used. The potential capacity of the one liquefier seemed adequate to supply the needs of several laboratories, allowing for reasonable shut-down times for servicing the machine. It also seemed reasonable to expect that the cost per litre of helium liquefied in bulk would be less than when liquefied on a small scale, and that this saving would offset costs of transport and of losses of liquid by evaporation during transport.

Further, the depreciation on the machine would be shared between several users, as would the mechanics' wages. Another important idea was that other established laboratories, capable themselves of producing liquid helium in bulk, could, if they wished, also enter the scheme by occasionally drawing on the N.P.L. supply in times of emergency (for example, if a liquefier broke down at an awkward moment). In return, they might possibly augment the main supply if the demand were temporarily too great for the N.P.L. to cope with. It is this aspect which eventually led to the scheme being called a 'Pool'. At the beginning there were, of course, many uncertainties. Nobody knew what would be the loss of liquid helium by evaporation during, say, a 200 mile train journey; there were no Dewar vessels manufactured in this country and there was not enough helium gas available. Above all, the potential demand could only be guessed at very vaguely.

The prime movers at this stage were the late Sir

Francis Simon and Dr. N. Kurti of the Clarendon Laboratory, Oxford, and the present writer, together with Dr. B. W. Robinson, Dr. R. W. Powell and Dr. A. R. Meetham of the Physics Division of the N.P.L. A meeting of those likely to be interested in the scheme showed that it was sure of support. Permission to use the liquefier for the new purpose was readily granted by the European Defence Council administration. The ways in which the organisational and technical difficulties were overcome can be gathered from later sections of this article. The first regular deliveries were made in April, 1955, and they have been continued with few breaks ever since.

No special formal arrangements have been made for the supply and distribution of liquid hydrogen. Any design of Dewar for use with liquid helium will certainly be suitable with liquid hydrogen, and there are several laboratories up and down the country where hydrogen is liquefied on a large scale.

Liquefaction and Distribution

In this section the equipment used in liquefaction and distribution will be briefly described.

The liquefier is of the pattern which was standard at the time it was supplied, see Figs. 1, 2. It produces its cooling by making the helium do external work driving an engine, and then undergoing a Joule-Thomson expansion. It liquefies about $3\frac{1}{2}$ litres per hour. It is capable of being run for three days a week, making 50 litres, though it has never been required to make so much, up to the present. A second compressor has recently been installed which can either be used as a stand-by or else can be run in parallel with the other so as to increase the output of liquid. One man is employed full time in running it, with occasional senior supervision. The transport vessels can be raised into position by a hydraulic lift. The liquid is transferred out of the liquefier through a vacuum-jacketed syphon and between 80% and 85% of the liquid made can be successfully transferred. The quantity of gas purified and handled per week is considerable.

Of the items of equipment that any regular user of the Pool must provide himself with, the first is obviously the transport Dewar. It was fortunate that Dr. J. M. Lock at the Radar Research Establishment, Great Malvern, had already designed and tested an efficient vessel which could be manufactured commercially. One can be seen in Fig. 3; they can also be protected in carrying cases so that they then resemble the American Dewars, one of which can be seen in Fig. 4. Each vessel is a helium Dewar inside a liquid nitrogen Dewar, all constructed of copper spinnings with necks made of alloys of low thermal conductivity. Radiation across the interspace is cut down to a small value by virtue of the T^4 law, and the inner sphere is highly polished to increase reflectivity. The principal cause of heat leak into the inner sphere is conduction down the neck, and this is reduced in two ways. Firstly, the upper part of the neck is made single walled and is arranged to be immersed in liquid nitrogen; thus the temperature gradient down the neck is greatly reduced. Secondly, the tube is long enough for the cold evaporating gas to exchange heat with the walls and this further reduces the heat influx.

Vessels holding 16 litres of liquid are available. They have the following characteristics: 12 litres of liquid nitrogen are initially needed to cool the outer vessel to liquid nitrogen temperatures and then a further 16 litres to fill it up. It has to be replenished about once every seven days. About $2\frac{1}{2}$ litres of liquid helium have to be syphoned in initially, to cool the inner vessel from liquid nitrogen to helium temperatures. When filled, the rate of evaporation averages about $1\frac{1}{4}$ litres per week. On a journey, the loss rate goes up by a factor varying from 5 to 10, but even then only about 500 c.c. at most are lost during six hours of travelling. The weight of the Dewar when full is about 1 cwt. These Dewars are also suitable for use with liquid hydrogen. It is well known that the main cause of evaporation of this liquid is the heat of conversion from ortho- to para-hydrogen. Assuming that the liquid has been fully converted before transfer, the loss rate is calculated to be only a few cubic centimetres per day.

It is important that a Dewar containing liquid should not be allowed to stand open to the air for too long, since there is the danger of solid air condensing in the neck and causing a blockage. The easiest way to prevent this is to close the outlet with a Bunsen valve—a piece of rubber tubing with a longitudinal slit in it—or with a valve from a gas mask.

Up to the present the Dewars have stood up well to the rough experience of transport by road and rail. Only once has one ever arrived damaged. Attempts to send them as registered articles, to ensure more gentle handling by railway porters, are usually unsuccessful because liquid-helium Dewars do not appear on the Schedule of Articles which can be registered. Liquid hydrogen may not be sent by rail. It would seem that Chief Constables have no objection to its being carried by road through their areas providing that it does not actually explode. So far, the use of rubber tubes to lead the evaporated gas safely away has enabled this requirement to be complied with.

One fact which has had an important influence on

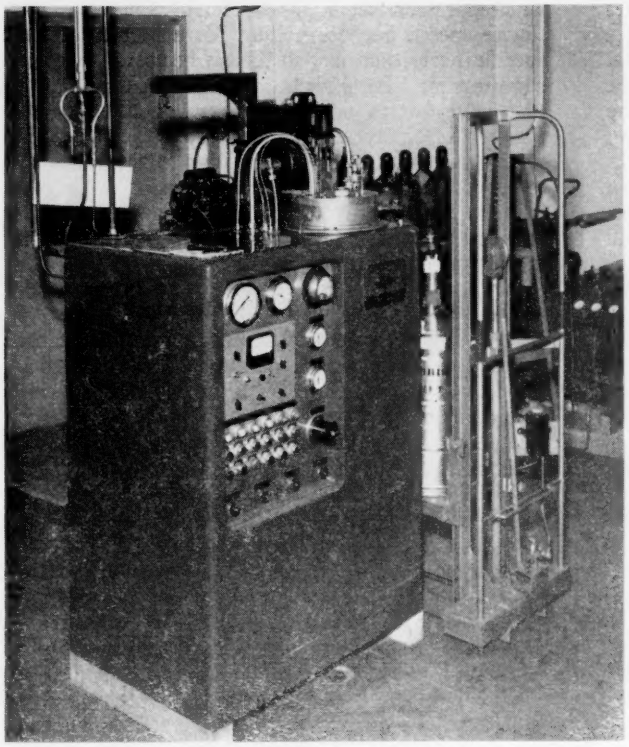

Fig. 1. The Collins helium liquefier at the National Physical Laboratory. The helium is made to drive an engine which does external work against the electric motor seen on top of the liquefier. The compressor is visible at the extreme right. A helium Dewar is in position on a hydraulic lift ready for filling.

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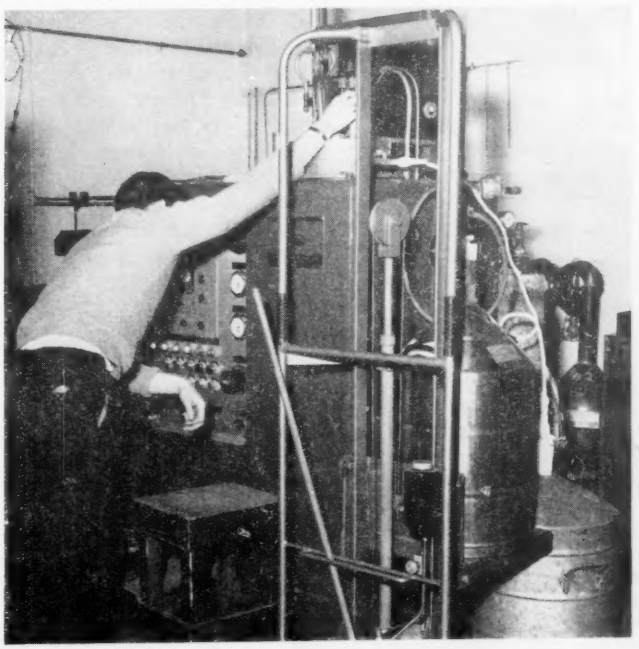

Fig. 2. Another view of the Collins liquefier. The helium Dewar is on the left; a second Dewar, without carrying case, can be seen in the background. The Dewar in the middle is feeding liquid oxygen continuously into the liquefier.

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the Scheme is that practically all the helium gas has to be specially imported from the U.S.A. It is a by-product from the oil wells and is so cheap there that it is used as an inert atmosphere for welding. But the transport costs to bring the heavy cylinders across the Atlantic put the price in this country up to about £25 per cylinder of 220 cu. ft. N.T.P. Therefore it, is important that each user should collect as much of the evaporated helium gas as possible for subsequent re-liquefaction. To encourage this, the liquid helium is sold at a certain price per litre, and then a substantial refund is given for gas returned to the N.P.L. The refund depends critically on the state of purity of the gas received. For collecting and transporting the gas, each user needs some sort of gasholder, a compressor and a small number of steel cylinders.

The gasholders present a minor problem at present. One litre of liquid helium when evaporated produces 3/4 cubic metre, *i.e.* rather more than 20 cu. ft. of gas. A metal gasholder to accommodate this, designed to have a small dead volume, is a bulky and expensive piece of equipment. Rubber gasholders (see Fig. 5) are cheaper, but by a peculiar process are quite permeable to oxygen so that gas stored in them becomes contaminated.

As a temporary measure, collapsible gasbags holding about 1 cubic metre, made of 0.007 inch polythene sheet, have been made in small numbers by Bristol University Physics Department for use in connection with the Pool. One can be seen in Fig. 6. They are made by the heat sealing methods used for balloons for cosmic ray research. They were sold at cost price, about one-twentieth of that of metal gasholders of similar volume. They are of a convenient size for carrying about from room to room and can, if necessary, be hung from the ceiling to save floor space. But after being dragged too often over dirty floors they tend to become perforated, and in any case the polythene is sufficiently permeable to helium to give a noticeable loss overnight. An alternative gas bag has been described by Squire[6] consisting of a double wall of Neoprene, the two layers of which are supported by a thin aluminium foil coated on both sides with petroleum jelly.

It is quite expensive to send cylinders by rail, and the manhandling of heavy cylinders at the N.P.L. is a job which involves a good deal of labour. On both counts it is desirable to economise by pumping the helium up to as high a pressure as possible. 30 atmospheres is a reasonable figure, 120 atmospheres much better. But, of course, the higher pressure requires a compressor with more stages. Usually the compressor is the most expensive piece of equipment. Small modifications are needed to standard types of air compressor (see Fig. 5). They must be run at slower speeds because helium has a higher ratio of specific heats than air, so that it heats up more on compression; the gaskets must be leak-tight, and it is advisable to collect any gas which blows out through the safety valves. Ordinary oxygen cylinders can be used with standard reducing valves. There is some confusion about the colour coding. English helium cylinders are painted brown all over; American ones are black with brown shoulder. The American convention is tending to be adopted.

Equipment Required for Utilisation

The equipment needed in connection with actual experiments with liquid helium will now be described. An article dealing with the installation in one laboratory of this kind has recently been published[7]; this section is therefore intended merely to supplement that article.

The Dewars which hold small quantities of liquid for experimental purposes are always 'home made'. Though not a major item of expenditure, they are very important of course, and often insufficient attention is given to their design. The inner Dewar usually holds 1-2 litres of liquid, and is protected by an outer Dewar containing liquid air, nitrogen or oxygen. These vessels can be made of metal or glass. For many purposes metal Dewars are superior—they can be made leak-tight by a competent workshop—but they suffer from the obvious disadvantage that the liquid cannot be seen. The greatest difficulty in making glass Dewars is presented by the dimensions of the tubing. It is difficult to obtain tubing of the right diameter and a reasonable wall thickness. Four separated concentric tubes have to be inserted inside one another to make a nest of two Dewars, and thick-walled tubing makes a clumsy looking assembly. Till recent years most tubing was drawn by hand, and since it was of variable diameter it was always possible to find just the right piece for any job. But nowadays the tubing is all moulded into a few standard sizes. Some sort of technique for 'stretching' the glass—for example, by passing it over heated bullet-shaped carbon mandrels—is essential for making tubes of the right diameters in order to obtain neat assemblies.

There is little doubt that the evaporation rate of liquid helium stored in long Dewars is far less than when stored in short Dewars. A Dewar 4 feet long with a small amount of liquid at the bottom keeps it much longer than one only 18 inches long. The rate of evaporation is markedly reduced if radiation from room temperature is prevented from reaching the liquid helium. There are two precautions, see Fig. 6. The first is to imitate the construction of the big metal

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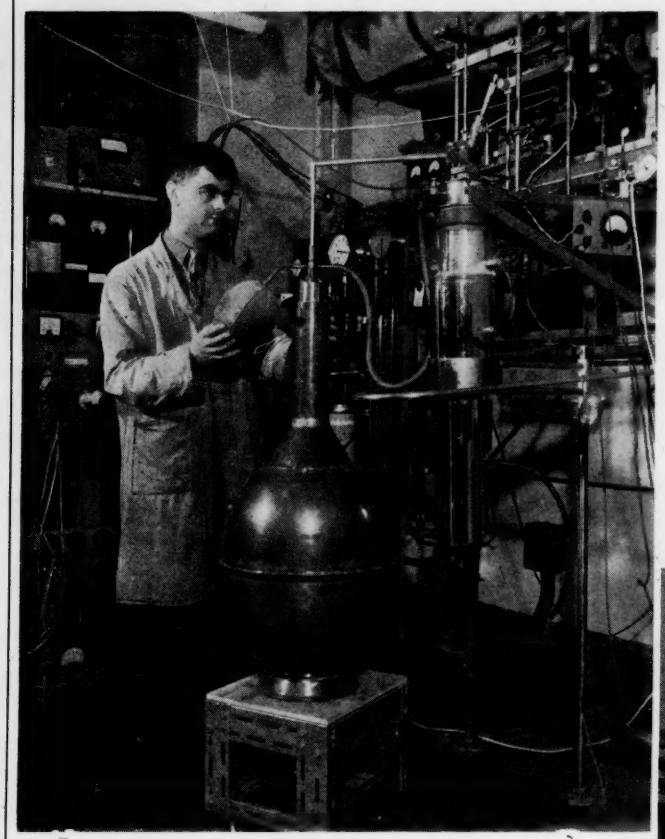

Fig. 3. Filling an experimental Dewar from the transport Dewar. (Services Electronics Research Laboratory, Baldock). The Dewar is made in this country. The syphon is metal. The liquid is pushed over by squeezing on a football bladder connected to the space above the liquid. The experimental helium Dewar is protected by a separate nitrogen Dewar. A rubber gasbag can be seen at the top of the picture. The electronic apparatus is all concerned with the measurements.

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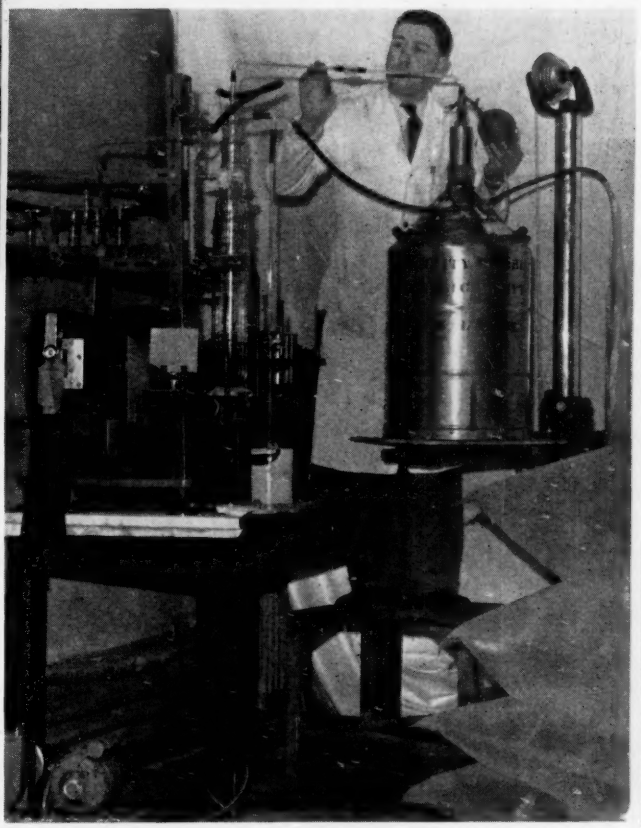

Fig. 4. Filling an experimental Dewar from a transport Dewar (Bristol University). The Dewar is American. Two L-shaped syphons are being used and the joint is clearly seen. The experimental helium and nitrogen Dewars are of an all-in-one construction. A polythene gasbag is seen at the bottom right. The experiment is a study of optical absorption in crystals at low temperatures—the spectrometer is covered with black cloth.

by courtesy of H. H. Wills Physical Laboratory.

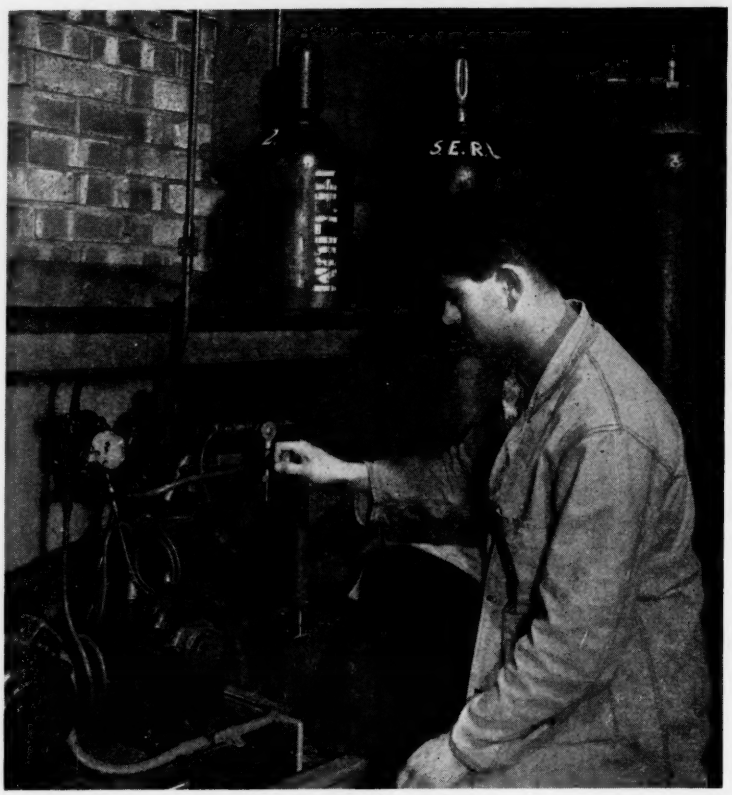

Fig. 5. The small single-stage compressor at Services Electronics Research Laboratory Baldock, used for compressing the evaporated helium gas into cylinders at 30 atmospheres pressure.

Fig. 6 (*below left*). All-in-one glass Dewars for liquid helium and liquid nitrogen. *Legend:* A—greased ground glass stopper for liquid helium inlet. B—tube for pumping on liquid helium. C—outlet for nitrogen vapour. D—liquid nitrogen. E—single-walled part of helium Dewar. F—double-walled part. G—liquid helium level.

Fig. 7 (*below*). Method of joining glass syphons. In the upper drawing, AA are the ends of the syphons; B is a short length of small-diameter rubber tubing closed with a glass stopper C; D. is the glass sleeve with folded-back lengths of rubber tubing EE in position. The lower drawing shows the syphons joined together ready for a transfer.

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Fig. 5

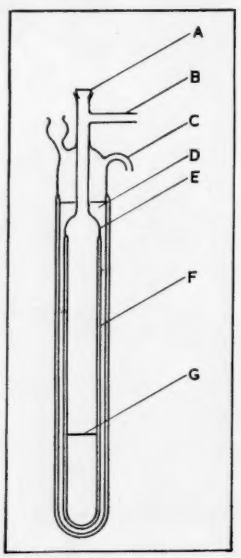

Fig. 6

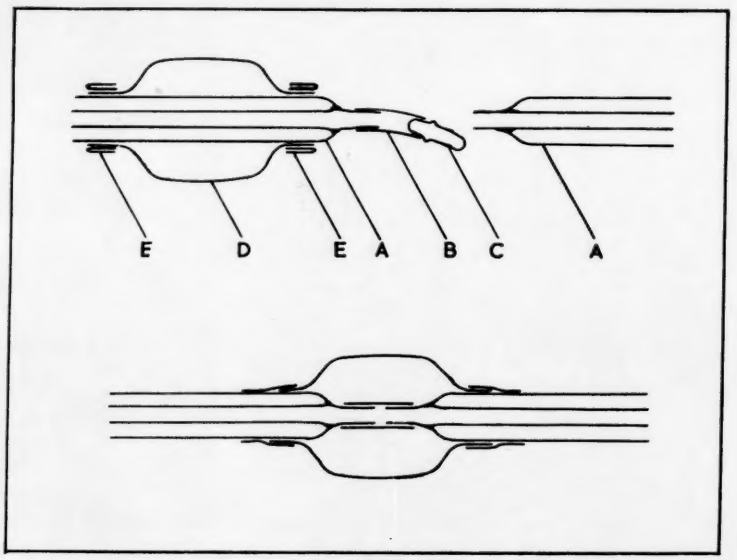

Fig. 7

storage Dewars, to make the upper part of the liquid helium Dewar single-walled, and to keep this always immersed under liquid air—this reduces the radiation from the upper part. The second is to introduce an unjacketed vessel containing liquid air at a suitable point in the upper part of the liquid helium Dewar. Polished metal radiation shields and the like seem to have little effect.

Borosilicate glasses such as Pyrex are comparatively highly permeable to helium. The mechanism is one of diffusion through the relatively large holes in the disordered crystal structure. It is the warm gas which diffuses through, not the liquid. The diffusion rate at liquid air temperatures is smaller than that at room temperature, by a factor of 10^4. Provided helium gas at room temperature is never allowed to come in contact with the double walled part of a Pyrex Dewar, it can be left sealed off. It is useful to leave 1 mm.Hg of air in the inter-space; this is sufficiently conducting to allow the contents of the helium flask (the calorimeter, etc.) to cool to liquid air temperatures initially, but when the liquid helium is syphoned in, the air freezes and the vacuum in the inter-space hardens. Metals and glasses such as Monax are impermeable to helium because there are no large holes in the crystal lattices.

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For many experiments, leads and tubes have to be brought into the apparatus. They are at room temperature at one end and are immersed in the liquid helium at the other. It is natural to make these of low-conductivity alloys in order to reduce the evaporation. But it must be remembered that heat exchange takes place from the ascending cold vapour, and this greatly reduces the heat input into the liquid at the bottom, compared with that estimated from superficial considerations of thermal conduction. A copper rod, say $\frac{1}{8}$ inch diameter and 30 inches long, causes surprisingly little extra evaporation, particularly if baffles are fixed to it to improve the heat exchange.

One phenomenon of profound interest to any experimenter has come into prominence in recent years: A vertical gas column with a negative temperature gradient in a downward direction may exhibit violent longitudinal oscillations[5,7]. The earliest recorded symptoms were that certain flasks lost helium very rapidly. Some flasks are even impossible to fill properly, although the inter-spaces are highly evacuated. The surface of the liquid is then seen to be violently agitated and low frequency oscillations can be detected with a rubber diaphragm communicating with the vapour space. Liquid hydrogen Dewars can sometimes be heard to be gently humming. The phenomenon is not properly understood, and several mechanisms have been suggested to explain it. It is possible sometimes to cure the oscillations by altering the resonant volume with a large cylinder connected to the vapour space, or by introducing damping in various ways, or by changing the temperature gradient. This may be one of the functions of the liquid air vessel above the liquid helium in Fig. 6.

Up to now, the design of experimental Dewars has been a matter of lore and experience. Many experimenters would welcome standard articles, commercially manufactured and properly designed. The idea of using plastics at low temperatures has been proposed[8].

To fill the experimental Dewar from the storage and transport vessel, a vacuum-jacketed syphon is needed. Many types of construction are described in the literature[9,10]. Most syphons are made U-shaped with one limb long enough to reach to the bottom of the transport Dewar. The main variations of design are associated with the type of spacer used for centering the inner tube within the outer.

Two L-shaped syphons which can be joined together are much simpler to handle. Soft black rubber tubing of the kind used for blood transfusions can be used for the joint; it has no filler in its composition and does not crack on cooling. The difficulty is to avoid the heat inflows which can occur at the junction. For liquid hydrogen it is sufficient simply to wrap cotton wool round it, to stop liquid air condensing on it. For liquid helium, a form of vacuum jacketing is needed with a path of high thermal resistance between the rubber joint and room temperature. If the syphons are of metal, the design is complicated[11]. A very simple technique is used with glass syphons by Dr. L. C. Jackson and his group at Bristol University (Figs. 4, 7). The glass sleeve is initially slipped over the horizontal limb of one syphon; after the joint has been made the sleeve is slid back over it and the rubber sleeves slipped into position. When liquid helium goes through the syphon, the air in the glass sleeve freezes, making a good vacuum jacket. Losses on transfer are no larger than when U-shaped syphons are used.

Pyrex syphons require re-pumping every time, however. After transfer, it is advisable to remove syphons of all types because the gas in them may go into oscillation.

The other apparatus needed in the laboratory to utilise the liquid helium—pump, manometer, level gauge, etc.—are adequately dealt with in the article cited[7]. It is to be hoped that with the increased use of liquid helium, some of these will eventually be manufactured commercially.

Table I. Capital Expenditure

Equipment required for Pool Service
Two Alternative Examples

(*a*)	*Appr. Cost* £	(*b*)	*Appr. Cost* £
Dewar Vessel	= 105	Dewar Vessel	= 105
Compressor (30 at.)	= 150	Compressor (120 at.) and 2 Cylinders	= 350
8 Cylinders	= 120	Hydraulic Lift to raise Dewar 6 ft. from Ground	= 60
Hydraulic Lift to raise Dewar 6 ft. from Ground	= 60		
Total	£435	*Total*	£515

Additional Equipment Required
in Laboratory to Facilitate Experiments

	£
Rotary Pump	= 130
Gas Meter	= 60
Total	£190

(*a*) *Grand Total* = £625 (*b*) *Grand Total* = £705

All prices valid for the period March 1956/1957.

Finance

It remains to try and give an account of the cost involved in using liquid helium made available by the Pool.

Approximate costs, divided into capital expenditure and running expenses, are given in tabular form (see Table I and II).

Table I gives two alternative sets of equipment required for obtaining and transporting liquid helium available from the Pool, and in addition, the minimum plant required to carry out experimental work in the laboratory.

The rotary pump must be fairly fast and is needed for pumping the cryostat to reduce the bath temperature; the gas meter is a useful instrument for monitoring the evaporation from both transport Dewar and experimental Dewar.

The running expenses shown in Table II are very largely governed by the directives issued by the Secretary of the Department of Scientific and Industrial Research, which state that the scheme must be financially self-supporting. This has been interpreted to mean that there must be no profit or loss over a period of the first 2-3 years. In addition, it is stipulated that the non-Government user should be charged the full economic costs, including depreciation, interest, import duties, etc., whereas the Government laboratories are exempt from these charges.

Of the prices shown for a litre of liquid helium in Table II labour only costs about sh. 14 per litre. Some adjustment will have to be made retrospectively to balance the accounts. The refund for helium gas returned to the Pool depends on the purity of the gas as shown in Table II. The amount shown as an average in that table is a mean taken over several users during the first year of working the scheme. The transport costs are based on the equipment shown in Table I (*b*) and allow for an amount of helium gas collected which fills rather more than one cylinder to 120 atmospheres. On the assumptions made in Table II the total rail and road charges would be about £7, resulting in sh. 14 per litre liquid helium.

Depreciation cannot properly be estimated yet, as there is no way of assessing the life of the equipment employed. The Dewar should be the only vulnerable piece of equipment.

It has been accepted that users of very small quantities of liquid—say 5 litres every 3 months—might be allowed to make no attempt to recover the helium gas, but to have to pay more for the liquid. The capital expenditure for such a user would then be only the £105 for the Dewar. No one has yet operated in this way.

The above layout refers to the cost of liquid helium per litre. Of course, a more realistic quantity in practice would be the cost involved per experiment. However, it is not possible to give a general estimate of how much helium is needed to perform an experiment since there are so many variables. For example, the losses during the transfer into the experimental Dewar are dominated by the heat capacity of the specimen to be cooled and in some bad cases a litre per run may be wasted in this way. Again, some experiments can be performed in a few minutes while others take several hours during which the liquid is boiled off at a great rate by electrical or mechanical power. The writer has taken part in experiments which have consumed less than a litre each, while others, lasting three days and nights without a break, have needed a full 10-litre consignment.

Comparison with Other Methods

An experimenter who wishes to start a new group working with low temperatures must compare the advantages of using the Pool with those of liquefying helium himself. He can choose between having a large liquefier, comparable in output with the Collins machine, or of operating on the small scale with miniature liquefiers. Detailed comparisons would be beyond the scope of this article and only the most important aspects will be mentioned here.

The attractive features of the use of the Pool are firstly that hardly any skilled assistance is needed in the laboratory—the only chores are the compression of the evaporated gas and the despatch of Dewar and cylinders. Secondly there is comparatively little investment of capital or effort which might later generate a resistance to leave low temperature research should the real interest of a problem shift elsewhere. Above all, the experimenter does not have to expend any effort on liquefying the helium; all his mental energy can go into the experiment itself. It is difficult to visualise much further simplification in the handling of the liquid and the production of the low temperatures.

On the other hand, the possessor of a large liquefier can feel independent of the help of others—though there is no reason to expect a new laboratory to be any less prone to breakdowns or accidents than the N.P.L. It would be particularly helpful if a machine were to be manufactured in this country for a reasonable price. Till that time, however, only a very large institution would find it cheaper to operate their own liquefier than to rely on Pool helium, particularly as a full time mechanic is needed to run such a liquefier.

Miniature liquefiers capable of making a few tens or hundreds of cubic centimetres of liquid helium are extensively used. The liquid is made and used in the one flask. The latest of these machines are beautifully designed[12 13] and the use that a skilled experimenter can make of a tiny amount of liquid is extraordinary. Indeed the very limitations of the techniques can present their own challenge and stimulus. But compared with the use of bulk liquid the small scale liquefaction demands much skill and experience and is quite complicated.

Conclusions

The use of liquid helium opens up research at low temperatures. Thirty-five years ago there was only one laboratory in the world where liquid helium could be produced. It is now available for research in scores of laboratories but the liquefiers are either costly to buy, or not easy to build. The Liquid Helium Pool can bring low temperatures to many more laboratories in this country because the equipment and techniques required are much less specialised.

The present price of producing and distributing the liquid is reasonable, but it would be markedly reduced if more users made use of the scheme.

Very low temperatures play an important part in many problems and it is to be hoped that the Liquid Helium Pool will be effective in furthering this kind of research.

Table II. Running Expenses

Government User £ s. d.		*Non-Government User* £ s. d.
3 0 0	*Price* of 1 Litre Liquid Helium at Teddington =	3 0 0
	Charge to cover Depreciation, Interest, Import Duties etc. =	1 0 0
14 0	*Transport* of a 10-litre consignment to a consumer 100 miles from Teddington and returned as gas in one 120-at. Cylinder per litre liquid helium =	14 0
£3 14 0		£4 14 0
1 0 0	*Refund* for Helium Gas returned to Teddington, the price varying from 1/3 per ft.^{3} at 98% purity to 9d. at 90% purity. Average established by experience =	1 0 0
£2 14 0	Total Net Running Cost per Litre Liquid Helium =	£3 14 0

All prices valid for the period March 1956/1957.

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January, 1957.

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LETTERS TO THE EDITOR

Pumping Speed Fluctuations in the Oil Diffusion Pump*

Sommaire

LES FLUCTUATIONS de pression dans les pompes à diffusion, du coté vide moléculaire et du coté vide primaire peuvent avoir de l'importance dans la détection des fuites. On donne des détails et les résultats des mesures faites sur ces fluctuations de pression qui dependent de la puissance de chauffage d'une pompe à diffusion à huile de 3 inches, equipée de trois différents types d'injecteurs. Les causes de ces fluctuations de pression, observées avec plus ou moins de puissance de chauffage sont discutées.

Introduction

THE FOLLOWING MEASUREMENTS on both the high vacuum and backing side of a small diffusion pump have shown the fluctuations in pressure to be dependent upon the heat input to the pump boiler. This is of considerable importance in leak detection work, where small changes in pressure must be detected. These experiments have been carried out on a single-stage oil diffusion pump of conventional design, having a diameter of approximately three inches with the two slightly varying jet constructions A and B shown in Fig. 1. Results are given in this paper for three jets: (1) Type A with a jet-to-wall distance d (see Fig. 1) of 5/16 inch, and (2) and (3) type B with jet-to-wall distances of 5/16 inch and 1/16 inch respectively. The experimental pump evacuated a small volume of approximately 0.2 litres and was 'backed' with a Metrovac 03B diffusion pump and a small two-stage mechanical pump in cascade. Pressure fluctuations on both the high vacuum and backing side were measured with thermionic cathode ionisation gauges.**

Experimental Procedure and Results

It was found difficult to describe the pressure fluctuations for any given conditions because of their essentially random nature. In every observation they were found to be made up of random changes occurring at intervals of between 1 and 10 seconds, very similar to those shown in Fig. 7 of the publication quoted. In order to make a qualitative comparison the fluctuations were defined arbitrarily as *the average*, taken over ten minutes, of the maximum pressure change in each individual minute. With this definition repeatable measurements were obtained to $\pm 20\%$ for any given operating condition.

At frequent intervals throughout the course of the work a check was made of any instability of either the flow of gas through the needle valve or in the ionisation gauge recording mechanism. The fluctuations in pressure, measured with the experimental pump cold, were always found to be less than 0.2% of the total gas pressure which represents the maximum total instability of the control and recording mechanism.

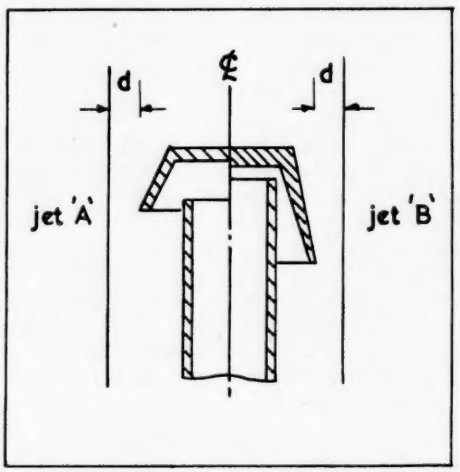

Fig. 1. Schematic diagram of jet types A and B.

Experiments with various jets showed the fluctuations to depend on the design of the jet and also on the rate of oil vapour circulation. In every case however the fluctuation $\triangle p$ was found to be directly proportional to the gas pressure, within the limits of experimental error. For example, for a pump with a jet-wall gap of 5/16 inch (Pump No. 2) values of $\triangle p/p$ of 0.005, 0.005, 0.007 and 0.006 were obtained for gas pressures of 1.0, 3.7, 6.5 and 10×10^{-5} mm. Hg respectively with a heat input of 320 watt to the boiler. In making these measurements, as in the rest of the work, either solid carbon-dioxide or liquid air was used in the trap in order to reduce the ultimate

* A report on work conducted in the Department of Electrical Engineering at the University of Liverpool.

** On the backing side the effect of any instability of the 03B pump was made negligible by throttling its speed down to 1 l./sec. Gas was admitted to the high vacuum side of the experimental pump through a small needle valve.

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pressure to the order of 10^{-6} mm. Hg. In this way the fluctuation of the ultimate pressure (about 1 to 5% of the ultimate pressure) could be neglected throughout the normal gas pressure range 10^{-5} to 10^{-4} mm. Hg.

The dependence of $\triangle p/p$ upon the design of the jet system and its operation is shown in Fig. 2, where $\triangle p/p$, expressed as a percentage, is plotted as a function of the heat input to the pump, (and therefore as a function of the rate of oil circulation) for three different jets. The variation of pumping speed with heat input is also shown on the same graph. The close relation between fluctuation amplitude and speed, the former passing through a minimum and the latter through a maximum at the same wattage is typical of all the experimental results.

The curves were not affected by varying the condensing wall temperature through the range 15 to 45°C, nor by changing the boiler design. In one experiment the total quantity of oil and the free surface area of the boiler were reduced to 30% of the normal value without producing any noticeable change in the results.

All the above experiments were made with air or nitrogen which gave essentially the same characteristics. In addition a few measurements were made with hydrogen, and although the results differed in detail the general shape of the characteristics was unchanged. The fluctuations decreased with increasing speed but were always considerably higher than the corresponding values with nitrogen. The minimum, for example, could not be reduced below about 4% for these particular jets.

The pressure fluctuations on the backing side of the experimental pump are shown in Fig. 3. Here, because of the throughput of oil decomposition products and the low pumping speed, the ultimate pressure P_u and also the fluctuations of the ultimate pressure $\triangle P_u$ were high. Whereas on the fine side cold traps were employed only for operating convenience, *i.e.* to reduce the pump-down time, on the backing side a liquid air trap was always essential to reduce P_u and $\triangle P_u$ to a reasonable value. Typical values of $\triangle P_u$ are shown in Fig. 4 as a function of heater power with (1) a liquid air trap, (2) a solid carbon-dioxide trap, and (3) the trap at room temperature, the values of P_u at 200 and 800 watt being marked on the curves for each case.

Discussion

A simple analysis shows that with the small high vacuum space time constant—of less than 0.1 second in the present experiments—fluctuations of pressure in the high vacuum space follow closely the fluctuations of pumping speed, thus the ordinates in Fig. 2

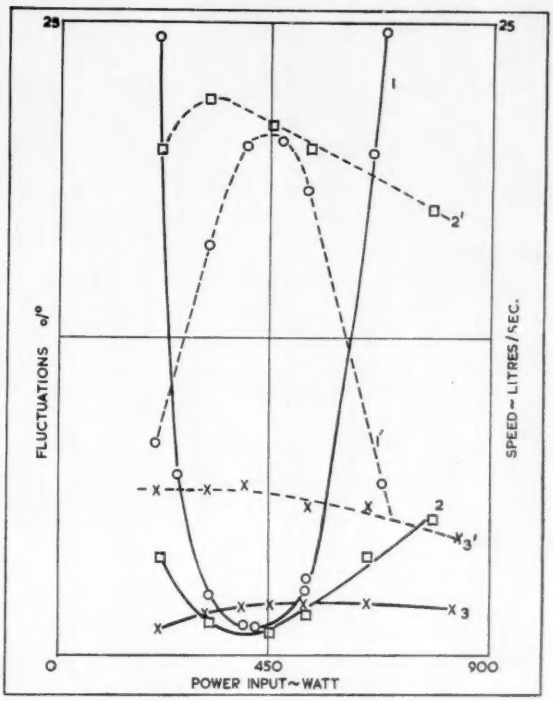
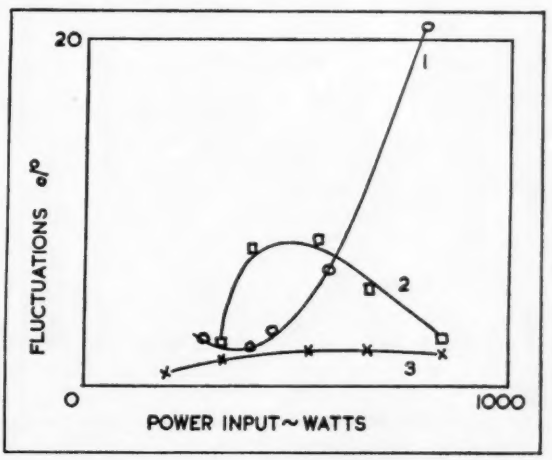


Fig. 2. Pressure fluctuations and pumping speed as a function of heat input for air. Curves 1 2 and 3 are the fluctuations for pumps numbered 1, 2 and 3 respectively. Curves 1′, 2′ and 3′ are the corresponding values of speed.

Fig. 3. Fluctuations of air pressure in the backing space as a function of the heat input to the pump. Curves 1, 2 and 3 refer to pumps 1, 2 and 3 respectively.



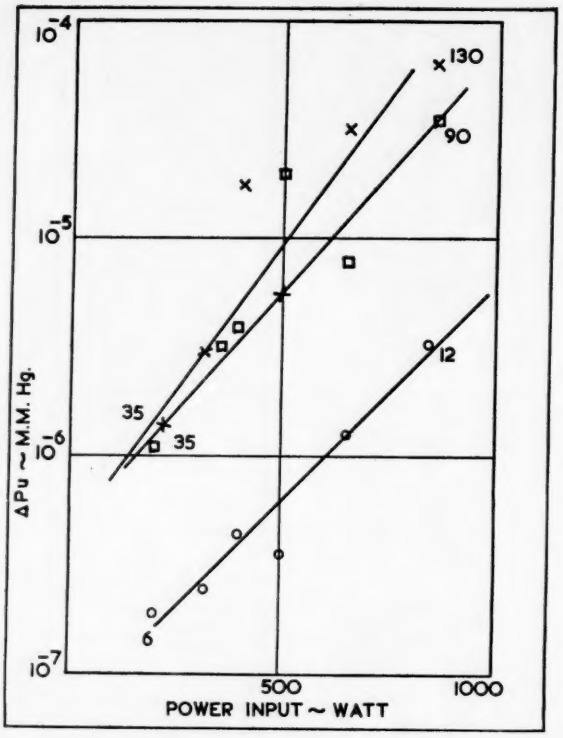


Fig. 4. Fluctuations of the ultimate pressure $\triangle P_u$ in the backing space as a function of the heat input to the pump. Values of the ultimate pressure $\times 10^{-6}$ mm. Hg are marked on each curve for heat input of 200 and 800 watt.

represent % speed fluctuations as well as % pressure fluctuations. These results show the speed to be most stable in the region of maximum speed, the instability increasing as the speed falls at both a high and a low heat input.

The speed falls as the heat input is reduced below the optimum because the gas molecules are more easily returned to the high vacuum from the backing space through the comparatively weak oil jet. The fluctuations of speed are almost certainly caused by instability of the oil jet, and associated with it, back-diffusion.

At a high heat input, where back-diffusion through the oil jet is quite negligible, the reduction in speed is caused by the inability of the gas molecules to enter the oil jet, some gas molecules being returned directly to the high vacuum by oil molecules travelling in opposition to the main stream. In this case an unstable oil vapour distribution causes the fluctuations in the pumping speed.

If the stability of the jet is, to a first approximation, independent of the oil vapour density, the fluctuations in speed must be proportional to the rate, at which the gas molecules are returned to the high vacuum space, *i.e.* proportional to the fall in speed from the maximum. Further confirmatory evidence of the relation between the fluctuations and the fall in speed at low wattage was obtained when the speed of pumping the backing space was increased from 1 to 50 l./sec. (by replacing the 03B with a pump having a speed of 200 l./sec.). This allowed the speed of the Jet (1) (see Fig. 2) to be maintained at the maximum down to 200 watt, and at the same time reduced the fluctuations to less than 2% at 200 watt. The difference in the characteristics at high wattage between two similar jet (see (1) and (2) in Fig. 2) is notable. These curves show how the pumping speed becomes critically dependent upon the jet design when operating above the normal heat input. This is probably the region where a true jet is being formed and the pump is taking on the characteristics of an ejector.

On the backing side the fluctuations are much greater than can be explained by the above simple theory for, because of the short time constant, the variations in gas throughput and therefore backing-side pressure should be negligibly small. The actual characteristics obtained (see Fig. 3) cannot be explained satisfactorily, especially curve (1) of Fig. 3, which is typical of the results obtained for jets of type A, in all of which the speed falls rapidly and the backing-side fluctuations become very large at high wattage. This must presumably be caused by some further instability in the jet or boiler.

When using a leak detector in the backing space it is probably best to operate the pump at a lower than normal heater power, *i.e.* with a speed only of the order of 75% of the maximum. A small heater wattage not only keeps the gas pressure fluctuations low (see Fig. 3) but also reduces the ultimate pressure (see Fig. 4). It is interesting to note that a solid carbon dioxide trap is not effective in condensing the products of decomposition of the pump oil. The ultimate pressure in the backing space can only be reduced effectively by cooling the trap to liquid air temperature.

It must be remembered that the values of fluctuations quoted here refer only to the case of the short time constant on both fine and backing sides of the pump, *i.e.* less than 1 second. When the fine side time constant increases above 1 second the fluctuations are, in effect, transferred from the fine to the backing side. An increase in the backing space time constant above 1 second would reduce the backing side

fluctuations without, of course, having any effect upon the fine side.

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A New Method of Sealing Off-Quartz Vessels in Vacuo

Sommaire

Normalement, les tubes de quartz sont fermés au moyen d'un chalumeau à mélange d'oxygène et d'hydrogène. Ce procédé a le disavantage de libérer de l'hydrogène qui se diffuse ensuite dans le quartz. On décrit une nouvelle méthode de fermeture sous vide au moyen d'un arc électrique, ainsi que l'outillage convenant à cette méthode.

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BECAUSE OF THE high melting point of quartz glass, about 1,800°C, the usual practice for manipulating quartz glass tubes is to use an oxygen-hydrogen or oxygen-acetylene flame. Furthermore, working with a torch supplied with these gases is not only tedious but also quite hazardous, unless great care is taken and first-class equipment is used.

However, the main disadvantage of this method is that thin-walled vessels of quartz are permeable to various gases, especially to hydrogen and helium, the permeability becoming greater as the temperature rises ([1]). There is always a possibility of free hydrogen being present when mixtures containing hydrogen or acetylene are used, partly because the proportions of the gas mixture are never exactly stoichiometric, and partly because water, which is one of the reaction products, dissociates at these high temperatures.

If we denote by q the volume of a gas in c.c. flowing through a wall of 1 cm² per second then $q=K\,P/d$. K is known as the permeability of the gas expressed in c.c. sec.$^{-1}$ cm^{-2} per mm. thickness d, at a pressure of P in cm. Hg.

Sometimes a more convenient unit $Q\mu_l$ is used which gives the amount of gas flow expressed in micron litres at 0°C per hour per cm² per mm. thickness for one atmosphere, hence $Q\mu_l=2{,}08.10^8K$.

For hydrogen $Q\mu_l$ is 1,33 at 900°C as compared with 0.198 for nitrogen, and rises very rapidly with temperature. The diffusing gases do not migrate directly from the gas phase, but after being adsorbed by the walls of the vessels. There are different mechanisms responsible for the migration of hydrogen, oxygen and other gases. At high temperatures, hydrogen passes through the lattice, whereas oxygen and nitrogen migrate mainly through slip planes. The permeability is therefore also proportional to the diffusion constant of the gas and to their respective solubilities in the material. The solubility of hydrogen in fused silica is about 0.01 cm.³ per cm.³ of gas at one atmosphere pressure for temperatures above 700°C.

For these reasons the vacuum obtained in vessels sealed off with a flame using a hydrogen or acetylene mixture is often inferior. This effect is, of course, troublesome only when the highest possible vacuum is required.

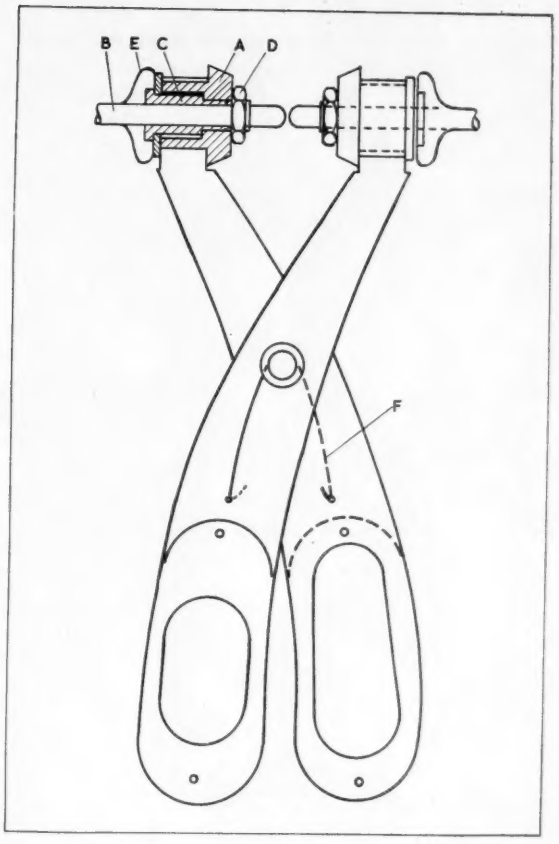

Fig. 1.

To seal off quartz vessels *in vacuo* a simple new method is described, which has proved its usefulness especially in sealing off evacuated mercury quartz burners. In this method the necessary high temperatures are obtained by an arc generated between two carbon rods. The power is supplied from an a.c. or d.c. source (normally from ordinary 220 volt a.c.) and adjusted with a suitable resistance to give a voltage drop of about 60 volt across the carbon rods.

The tool which is very simple in its design and can easily be made in any laboratory workshop is shown in Fig. 1. It is made from light metal and has the shape of tongs, the ends of which are fitted with porcelain tubes (A), to insulate the carbon rods (B) from the body of the tongs. Into these porcelain tubes are inserted copper tubes (C) of about 8 mm inside diameter; one end is flanged and the other threaded and slit to form a simple chuck. A nut (D) tightens the chuck around the carbon rods, which have nearly the same diameter as the copper tubes, thereby ensuring good electrical contact, and at the same time locates the copper tube firmly in the porcelain. The ends of the carbon rods remote from the chuck are supported by the copper springs (E). The leads from the power supply are soldered to the copper tubes and springs and fasten along the body of the tongs. To make arrangements even safer, insulated handles may be fitted to the tongs. The handles of the tongs are forced apart by the steel spring (F), which, by giving the tool a positive 'feel', facilitates the control of the length of the arc.

With the aid of this tool the sealing-off process takes about one minute. The eyes of the operator have of course to be protected by tinted glasses.

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15th September, 1956.

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BOOK REVIEW

Vacuum Deposition of Thin Films. By L. HOLLAND. (Chapman and Hall Ltd., London, 1956). Pp. 541. Price £3 10s. 0d.

Twenty years ago vacuum evaporation was just coming into use for the deposition of reflecting films of aluminium on glass while cathodic sputtering eked out a modest existence as the more traditional process for making metal films. Today it takes a book of 500 pages to summarise the special techniques which have been evolved for depositing almost any metal and many compounds—and for depositing them on materials which at one time would have been rigorously excluded from any vacuum system on the grounds that they were much too 'gassy'.

The advance of the science and application of thin films has been made by three agencies, each supporting and stimulating the others. These agencies are: Vacuum engineering, the technology of deposition and research into the structure and properties of films.

Mr. Holland's book is primarily concerned with the technology of thin film deposition but the reader who uses this book as something more than a guide to all the available methods will find not only a full chapter summarising the physics of film growth and structure, but many other references throughout the book to scientific work which has clarified the phenomena on which the various processes are based. The existence of this scientific background does not mean that vacuum processes and their products are fully understood. On the contrary, when the facts are gathered together and discussed in this way the gaps in the existing knowledge of the subject show up all the more clearly. This exposure of the underlying scientific problems should be of value to the many research workers now entering this new field.

It is incontestable that advances in vacuum engineering itself have had a great influence on the development of deposition techniques. The author has, however, resisted the temptation to describe in detail the construction of the whole range of vacuum apparatus. There are already several books on this subject and the necessity of constructing one's own apparatus is fortunately in most cases a thing of the past. It is nevertheless essential to know just what can be done with modern vacuum plant, especially in coping with the evolution of large volumes of gases and condensable vapours. The descriptions of the booster vapour pump, the gas-ballast pump, and

other innovations are therefore welcome. Turning now to the uses which are made of thin films on a commercial scale, it may be said that while a very wide range of applications has been found, there are five really outstanding products which have each absorbed a vast amount of technological effort. These are: aluminium films, optical interference filters, metal and other films on plastic materials, films for electrical purposes, and metal oxide films. It is not surprising that chapters on the preparation and properties of these films make up nearly half the book.

Aluminising, as has already been pointed out, is the oldest of the commercial evaporation processes. Indeed, most of the basic practical problems of successful film deposition—cleaning of the glass support, form of vapour source, attainment of the necessary vacuum in a demountable system, film thickness distribution, stability and strength of film—were encountered in the struggle to raise aluminising to the status of a reliable industrial process.

In addition to the work on the basic process, a great deal of attention has been paid to the improvement of the quality of aluminised mirrors by discovering the cause of granulation on surfaces of high curvature, by improving the bonding to the glass support, and by protecting the surface with an anodised or deposited film. These are the advances of greatest practical importance and they are given due prominence in this chapter, but they are accompanied by accounts of more modern investigations on a large number of other variables affecting reflectivity and durability. When the utmost efficiency is to be achieved, as for instance in maintaining high reflectivity in the ultra-violet, a familiarity with this more recent work is essential.

To those accustomed to thinking of surface coatings as belonging to the realm of scientific instruments it seems incredible that a large proportion of commercial vacuum plant is used for the surface coating of paper, textiles, plastics and lacquered metals. In particular, both plastics and dull metals may be made to glitter in a most attractive way by lacquering, aluminizing, and then lacquering again, this time with a tinted lacquer. The coating has to be done on a large scale for the sake of cheapness, and the technical problems have been considerable. It is at this point that a criticism of the arrangement of chapters must be voiced. The ordering of subjects is too haphazard and in parts perversely so: "The Degassing of Plastic Materials *In Vacuo*" forms Chapter 2 but one has to turn to Chapter 12 for "Evaporated Coatings on Plastic Materials and Lacquered Components". This book is much more than a book of reference and deserves a more coherent layout.

The study of optical interference films is a science in itself, and their exploitation has forced the thin film techniques to a further degree of refinement. The special requirement here is exactitude in the control of film thickness and great uniformity in deposition over large areas. Mr. Holland has done well in condensing the essentials of this subject (including a brief introduction to the theory of the various thin film combinations) into 50 very informative pages. Given the proper equipment, high efficiency beam-splitters and optical filters of many useful types can be constructed, though it must be recognised that interference filters still have their peculiar disadvantages, which include a comparatively high cost. The simpler films such as antireflection coatings and neutral beam-splitters are of course well established, and the Fabry-Perot filters deserve to be more widely known and used.

As readers of this journal will know, Mr. Holland has been active in exploiting the new application of sputtering in which reaction of a sputtering metal with oxygen is encouraged, resulting in the deposition of the metallic oxide. The preparation and properties of several oxide films by this method are described, and it is clear that the process lends itself to considerable development. Electrically conducting films made in this way are now coming into use for carrying heating current on glass windows without exacting a high price in absorbed light.

A separate chapter is devoted to the deposition of metals in high vacuum for electrical purposes. In cases such as the zinc coating of paper for capacitors this is carried out on a large scale and much work has gone into the development of the special plant.

The subjects which have been selected for discussion in this review occupy ten chapters. The other six chapters are concerned with the characteristics of vapour sources, the evaporation of a range of substances, the achievement of durability, cathode sputtering in general, and the art of shadow-casting and replica production.

Perhaps the most outstanding feature of this work is the wealth of detail assembled round each topic. Even so it has naturally been impossible to do more than summarise the information, but the very numerous references give every opportunity for closer study. These references are collected together at the end of the book, a much better arrangement than the more usual one of putting them at the end of each chapter.

Mr. Holland is to be congratulated on having had the courage to tackle this vast subject and on having brought it to so successful a conclusion.

K. M. GREENLAND.

. . . *of vacuum interest*

Lectures

United Kingdom. The Department of Electrical Engineering and Physics of THE ROTHERHAM COLLEGE OF TECHNOLOGY, Howard Street, Rotherham, Yorks., has announced a short full-time course on HIGH VACUUM TECHNOLOGY which is to start on Monday, July 1st and finish on Friday, July 5th. The course will cover the construction and maintenance of vacuum pumps and gauges, and will include practical work and visits to local industry. For further particulars contact the Registrar of the College.

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United States. The College of Engineering of THE NEW YORK UNIVERSITY are holding a course on VACUUM METALLURGY from Monday, June 10th to Friday, June 14th. Details of the programme are given as follows:

Vacuum Pumping Systems.
Lecturer : *H. A. Steinherz.*

Selection of Gauges, Leak Detection, and Trouble Shooting.
Lecturer: To be announced.

Thermodynamics and Kinetics in Vacuum Metallurgy and the Effect of the Pressure Variable.
Lecturer : *Thomas King.*

Thermodynamics and Kinetics of Induction Melting.
Lecturer : *A. M. Aksoy.*

Thermodynamics and Kinetics of Arc Melting.
Lecturer : *P. C. Rossin.*

Arc Phenomena, Basic and Applied.
Lecturer : *E. W. Johnson.*

Selection of Power Supply and Methods of Arc-Electrode Motion Control.
Lecturer : *E. J. Borrebach.*

Survey of European Arc Furnace Design.
Lecturer : To be announced.

Consumable-Electrode Arc Melting of Refractory Metals.
Lecturer : *R. M. Parke.*

Consumable-Electrode Arc Melting of Non-Refractory Metals.
Lecturer : *J. H. Johnston.*

Skull and Permanent Electrode Melting.
Lecturer : *John L. Ham.*

Induction Melting of Ingot Products.
Lecturer : *W. Jones.*

Induction Melting of Cast Shapes.
Lecturer : *W. Beamer.*

Degassing in the Molten State.
Lecturer : *Max Auwärter.*

Degassing in the Solid State—Use of Vacuum Furnaces in Degassing, Brazing, Annealing, etc.
Lecturer : *R. F. Gunow.*

Vacuum Reduction and Distillation.
Lecturer : *H. W. St. Clair.*

High Temperature Alloys—Cast and Wrought.
Lecturer : *M. E. Cieslicki.*

Ferrous Base Alloys.
Lecturer : *A. M. Aksoy.*

Refractory Metals.
Lecturer : *Ivor Campbell.*

Nuclear Power Plant Materials.
Lecturer : *Arthur P. Beard.*

Applications in Electronic Materials.
Lecturer : *C. W. Horsting.*

Powder Metallurgical Products.
Lecturer : *H. H. Hausner.*

Vacuum Coating Processes.
Lecturer : *Philip J. Clough.*

Analysis Techniques.
Lecturer : *F. C. Benner.*

Future Horizons in Vacuum Metallurgy.
Lecturer : *J. M. Moore.*

The introductory address is given by Mr. R. S. Morse, President of National Research Corporation. For details please contact Dr. R. F. Bunshah, College of Engineering, New York University, University Heights, New York 53, N.Y.

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United Kingdom. The Department of Applied Physics of THE NORTHAMPTON POLYTECHNIC, St. John Street, London, E.C.1, has arranged for a series of lectures to be held on INDUSTRIAL APPLICATIONS OF HIGH VACUUM TECHNOLOGY, every Tuesday, commencing March 5th, 1957, at 7 p.m.

Details of the lectures are given as follows:

High Vacuum Technology in the Valve Industry.
Lecturer : *R. O. Jenkins.*

Glass-to-Metal Seals for High Vacuum Apparatus.
Lecturer : *O. Adams.*

Ultra-High Vacuum.
Lecturer : *W. Steckelmacher.*

New Pump Developments and Systems for Special Purposes.
Lecturer : *B. D. Power.*

Vacuum Metallurgy.
Lecturer : *M. E. Harper.*

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Vacuum Coating.
Lecturer : *L. Holland.*

Vacuum Drying.
Lecturer : *T. W. G. Rowe.*

Large High Vacuum Systems.
Lecturer : *E. J. Greer.*

FURTHER LECTURES are announced as follows:

Monday, March 11th 1957 at 6.0 p.m.

Vacuum Techniques.
Lecturer : *D. J. Taylor.*
Held at : Winter Gardens, Malvern, Worcs.
Arranged by : The Institution of Electrical Engineers, South Midland Centre.

Thursday, April 11th 1957 at 7.0 p.m.

Applications of Vacuum Techniques in Metallurgy.
Lecturer : *M. Harper.*
Held at : Liverpool Engineering Society, The Temple, 24 Dale Street, Liverpool.
Arranged by : The Liverpool Metallurgical Society.

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United States. THE COMMITTEE ON VACUUM TECHNIQUES has announced that the FOURTH ANNUAL SYMPOSIUM will be held in Boston, Massachusetts on October 9th-11th 1957 at The Hotel Somerset, Boston, Mass.

Belgium. L'ECOLE ROYALE MILITAIRE have announced a Conference to be held at Namur (Belgium) on Monday, 9th June to Thursday, 12th June, 1958 under the title of LE PREMIER CONGRES INTERNATIONAL POUR L'ETUDE DES TECHNIQUES DU VIDE. The programme will cover vacuum physics and scientific and industrial applications of vacuum engineering methods. For further information contact Professor Dr. E. Thomas, L'Ecole Royale Militaire, 30 Av. de la Renaissance, Bruxelles 4.

France. LA SOCIETE FRANCAISE DES INGENIEURS ET TECHNICIENS DU VIDE have organised a one-day conference to take place on Thursday, 16th May 1957, at 44 Rue de Rennes, Paris VI^{e}. During the morning session lectures will be given on Vacuum Evaporation Problems by Messrs. *Gervais*, *Heisinger* and *Dunoyer*. The afternoon session deals with Vacuum Metallurgy, and lectures will be contributed by Messrs. *Barrett*, *Meny*, *Ortel*, *Givord* and *Stohr*.

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United States. THE THIRD HIGH-TEMPERATURE MATERIALS CONFERENCE by THE AMERICAN INSTITUTE OF MECHANICAL ENGINEERS is being held on April 16th and 17th 1957 at Cleveland, Ohio. The programme includes a discussion on Vacuum Melting. For particulars, contact Dr. M. A. Steinberg, Horizons Inc., Cleveland, Ohio.

Italy. An INTERNATIONAL CONFERENCE ON IONISATION PHENOMENA IN GASES will be held in Venice on June 11th-15th 1957. The organiser is Prof. Ugo Facchini, Laboratori CISE, Via Procaccini 1, Milan.

Books

The following books on vacuum subjects have been published recently:

Germany.
Elektrische Vorgänge in Gasen und in Vakuum.
Author : *N. A. Kapzow.*
Published by : Deutscher Verlag der Wissenschaften, Berlin (1955).
Pp. 700. Price £3 10s. 0d.

United States.
Arcs in Inert Atmospheres and Vacuum (Symposium).
Edited by : *W. E. Kuhn.*
Published by : John Wiley and Sons, Inc., New York (1956).
Pp. 188. Price £2 14s. 0d.

Personal

United Kingdom. Dr. G. T. K. Conn, until recently Reader in Physics at Sheffield University, has been appointed to the Chair of Physics at the University of Exeter.

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United Kingdom. Dr. G. H. Metson, Member of the General Post Office Research Laboratories, London, known for his work on various aspects of the design and performance of long-life repeater valves, has been promoted to Deputy Chief Scientific Officer.

New Processes and Applications

Coating by Vacuum Evaporation. It is reported that the satellite to be launched by the United States during the present International Geophysical Year may carry a small strip of Nichrome about $\frac{1}{4}''$ wide, $1\frac{1}{2}''$ long and 0.0001 mm. thick on part of its surface.

The metal film is applied to glass by means of vacuum evaporation and is to serve as a means of measuring surface erosion caused by micro-meteorite impact during its flight. As the film becomes pitted by the impacts, its electrical resistance increases and this increase is recorded by suitable means.

Industrial

United States. P. J. Clough has been appointed Director of the Metallurgical Research Department of the National Research Corporation, Cambridge, Mass.

United States. The Crucible Steel Company of America has purchased the National Research Corporation interest in the Vacuum Metals Corporation. The latter has already been operating for several years as a joint subsidiary of both companies.

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Finland. The building of a Vanadium factory is reported. This is the first in Europe and will meet 50% of Western European needs, until now entirely dependent on American and South African supplies.

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VACUUM
CLASSIFIED ABSTRACTS

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Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

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Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract, *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

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10 — VACUUM ENGINEERING (GENERAL) — 10

High Vacuum Equipment

205/I

Russia. Vacuum apparatus and plant available in Russia at the present time is reviewed. The basic requirements of a high vacuum system are briefly discussed and details are given of the design and performance of the various types of pumps, gauges, seals and leak detection apparatus which are manufactured in that country.

Article by K. A. Savinskii *Zavodskaya Labor.* No. 9, 1955 Translated by R. Hardbottle *Vacuum* 4, July 1954 326-340

Sommaire: Information détaillée sur l'appareillage à vide disponible en Russie, en particulier, les pompes, jauges et appareils détecteurs de fuite.

High Vacuum Engineering

206/I

United Kingdom. The mean free path of air molecules at 760 mm. Hg pressure is 2×10^{-6} inch.; at 0.001 mm. Hg pressure the mean free path is approximately 2 inch. The term 'high' vacuum is normally applied to the case where the mean free path in the residual atmosphere of an evacuated vessel is larger than the dimensions of the vessel. Vacuum processing in industry requires pressures ranging from 1 mm. down to 10^{-6} mm. Hg. The article describes design features and performance data of three makes of mechanical pumps (Edwards, Metropolitan-Vickers and Kinney) employed in connection with the exhaustion of modern large-size industrial plant. Vacuum processing in industry is indicated where one or more of the three following requirements have to be met: (*a*) Operation in an inert atmosphere, (*b*) operation at reduced processing temperatures, and (*c*) unobstructed (linear) propagation of vaporised material. The following examples are quoted: (*a*) Certain metallurgical processes, (*b*) freeze drying, and (*c*) metallisation and short-path distillation. The basic formulae are given for the determination of pumping speed and evacuation rates. For instance, the time T (minutes) required to pump down a system of volume V (litres) with a backing pump having a speed S_p (litres per minute) from atmospheric pressure to 0.1 mm. Hg can be assessed by

$$T = 10\ V/S_p$$

Mechanical rotary oil-sealed pumps can be used directly for evacuation down to a fraction of 1 mm. Hg, and have a constant displacement which ranges from 20 to about 20,000 litres per minute according to type and model. The vane-type of pump consists of a stator immersed in an oil bath. A rotor fitted eccentrically inside the stator carries a pair of vanes pressing outwards against the inner surface of the stator. As the rotor revolves the gas collected by the pump on the inlet side is compressed to a pressure above atmosphere and then discharged through a non-return valve also submerged in the oil. In order to reduce the adverse effect of dead space at the discharge side oil is leaked into the compression chamber resulting in the oil mixing with the gas and carrying the gas with it when discharged through the exhaust valve. However, the oil serving as a sealing agent between vane and inner surface of the stator will carry a certain amount of dissolved gas and, as the oil is penetrating to the inlet side of the pump, the gas will be partially released into the vacuum system. The higher the compression ratio, the greater is the danger of gases being returned to the vacuum system. In order to overcome this difficulty, two-stage rotary pumps have been developed which may give an ultimate pressure as low as 0.1 micron Hg. The lowest pressure obtainable with rotary pumps is that of the vapour pressure of the oil and little can be gained by providing for more than two stages in a pump. Another serious limitation to the efficiency of rotary pumps is contamination, for instance by water vapour. Air at 20°C having a relative humidity of 50% contains about 9 gram. of water per cubic metre. To prevent water vapour from reaching the pump, traps filled with desiccants such as phosphorous pentoxide or potassium perchlorate are inserted in the pumping line. An alternative way is the use of freezing traps cooled with liquid air or solid carbon dioxide, and, finally, continuously refrigerated traps can be used at somewhat higher temperatures. A preferable solution is to allow the contaminants to dissolve in the oil, and to circulate the oil continuously through an external unit where the oil is heated and volatile contaminants flashed off. Details are given of an oil stripper developed for the purpose. Finally the rotating-plunger type of mechanical pump is described. Single-stage pumps of this kind give an ultimate pressure of 10 micron Hg. Two-stage models give an ultimate pressure of 0.5 micron Hg.

Article by A. S. Darling *Mech. World* August 1954 342-348

Sommaire: Un article détaillé sur l'appareillage à vide, donnant les performances et la construction de pompes méchaniques, en particulier de pompes à palettes et de pompes à cylindre.

Ultimate Vacuum in a Vacuum-Enclosed Ionisation Gauge

207/I

See Abstract No.: 143/II

Portable Laboratory Vacuum Pump

208/I

See Abstract No.: 139/II

The Measurement of the Pressure in the Determination of Pump Speed

209/I

Holland. There is no commonly agreed method of determining the speed of a pump. One noteworthy method was proposed by Dayton in 1948 but it has not been accepted generally. Contributions to the problem made by other workers are briefly reviewed followed by a close study of the basic elements of the definition of pressure.

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Special attention is given to the case where the number-density of the molecules varies along the mean free path. Such variations may occur at low pressures and exist, in particular, in the region of the pump mouth. The actual method of measuring pressure needs more consideration and it is shown that a gauge connected by a tube to the system records the number of molecules per unit area arriving at the gauge-end of the tube. The conditions at the pump mouth have been investigated and an interpretation is given of the results of some measurements with regard to the distribution of the incident molecules. In conclusion, a method of measuring the speed of a pump is proposed which differs from Dayton's method. The new method is based on well-known concepts in the physics of gas flow at very low pressures. It has been put forward before, but simply as a postulate.

Sommaire: On propose une méthode de mesure de la vitesse d'une pompe; cette méthode est un développement de celle conçue par Dayton en 1948.

Article by A. Venema *Vacuum* 4, July 1954 272-283

Sources, Measurement and Control of Backstreaming in Oil Vapour Vacuum Pumps 210/I

United Kingdom. In all vapour diffusion pumps working fluid vapour continuously backstreams through the pump inlet to an amount depending on the design and method of operating the pump, with the result that the vapour pressure in the pumped system is increased beyond that corresponding to the temperature of the pump-cooling water, and undesirable condensation of fluid in the system may ensue. The sources of the backstreaming vapour are studied and methods of control are discussed which do not adversely affect the pump performance. A critical review is made of various methods of measuring backstreaming, and the technique adopted by the authors is described. The lip of the top jet nozzle is shown to be an important source of backstreaming, and a qualitative explanation for this is put forward. Two new control devices are then described: One, a cooled 'guard ring' around the nozzle lip which intercepts the backstreaming vapour at source, and the other, a nozzle with a controlled temperature gradient along its wall which is intended to condense the randomly moving outer layer of the vapour jet from which the backstreaming molecules originate, and which is contrary to all present practice.

(*Authors*)

Sommaire: Les sources de remontée de vapeur dans les pompes à diffusion sont étudiées, des méthodes de contrôle n'affectant pas les performances des pompes sont discutées.

Article by B. D. Power & D. J. Crawley *Vacuum* 4, Oct. 1954 415-437

Performance Characteristics of Barium Getters with Particular Reference to Their Applications in Thermionic Valves 211/I

See Abstract No. : 81/IV

Applications of a Tracer to Cathode-Gettering and Gas-Adsorption Problems 212/I

Holland. It has been observed that the residual gas pressure in a cathode-ray tube decreases when the cathode is heated, even if there is no potential on the anode. This suggests that the barium-strontium oxide layer of the cathode exercises a gettering action. In order to verify this hypothesis the authors employed a method using a gaseous radioactive tracer. As carbon dioxide gas is known to have a poisonous effect on the oxide layer, the authors decided to prepare carbon dioxide with the radioactive ^{14}C–isotope. The latter emits a beta-radiation weak enough to be absorbed by the glass of the cathode-ray tube and has a half-life of 6,400 years. The tests were carried out in a 5-inch. oscilloscope tube fitted with a barium-strontium oxide cathode and a gun made of stainless steel, but one screening diaphragm was made of stainless steel stabilised by titanium. Details of the initial pumping and the subsequent cathode activation process are given. After sealing off the tube, a barium-aluminium getter was flashed giving an initial pressure as low as 10^{-6} mm. Hg. Subsequently $BaCO_3$, containing 1 μc. ^{14}C, held in a metal tube connected to the oscilloscope tube, was heated and $^{14}C^{16}O_2$ was produced. Measurement of the pressure immediately following this step indicated 9×10^{-6} mm. Hg. Then the cathode was heated with the anode current off for 30 minutes at 1,100°K. Finally the tube was dismantled and the activity of the various components measured by a Geiger-Müller counter. Due to the weak activities of the components each count was taken over a period of 10 minutes. The results of the experiment are shown in a table reproduced below. The outcome of a second experiment with another tube proved that the results were reproducible.

Measured Counts per mm² for Cathode, Diaphragm and Other Gun Components

Sample	*Specific activity (number of pulses after 10 min. counting time, per square millimetre of surface, without background)*
Barium-strontium oxide layer of the cathode*	51.00
Stainless steel, stabilised by titanium	0.48
Stainless steel	0.11

* As the surface of the cathode is very porous, part of the activity measured will originate from $^{14}C^{16}O_2$ in the pores. The radiation of these ^{14}C atoms, however, will have been absorbed partly by the oxide layer.

Sommaire: Description d'une méthode et de ses résultats, permettant de vérifier les qualités getter de la couche barium-strontium des cathodes à oxyde, dans les tubes à rayons cathodiques.

Letter by F. de Boer & W. F. Niklas *Brit. J. Appl. Phys.* 5, Sept. 1954 341-342

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Manipulation of Radioactive Gases in High Vacuum Apparatus 213/I

See Abstract No. : 186/III

The Production of Radio-Isotopes 214/I

See Abstract No. : 187/III

Metallurgical Trends of Interest to Chemical Engineers 215/I

United Kingdom. A review is given of new developments in metallurgy for the benefit of the chemical engineer, with particular reference to metals employed in atomic energy plant. Details are given of the extraction and preparation of uranium for use in atomic piles, where fluorine chemistry plays an important part. After its reduction to metal the uranium is melted and cast under vacuum into bars of dimensions suitable for canning. Since the natural uranium contains no more than 0.7% of the fissile isotope, special methods have been developed to enrich the metal, *i.e.* to increase its fissile isotope content. The method used for the purpose in the United States and the United Kingdom is the diffusion process whereby the lighter, fissile isotope, ^{235}U, diffuses through a membrane and, by using a number of stages, enriched metal is obtained. The process itself is not carried out in vacuum but the plant requires leak tightness satisfying high vacuum standards, a task of considerable magnitude considering the plant consists of miles of pipes and thousands of joints. The author proceeds with a discussion of production characteristics and properties of titanium, zirconium, tantalum, niobium and molybdenum. In the case of titanium, reference is made to the Kroll process developed under the auspices of the U.S. Bureau of Mines. Pure titanium metal is weak and of little commercial value. It is preferably used in alloyed form. For instance, the titanium alloy containing 2.7% chromium, 1.3% iron, 0.25% oxygen, 0.02% nitrogen, less than 0.03% tungsten and 0.02% carbon has an ultimate tensile strength in the annealed state ranging from 62.5 to 74 tons per square inch. Titanium has high corrosion resistance but tends to become brittle when exposed to temperatures above 550°C because of the absorption of oxygen and nitrogen. Titanium alloys cannot be used in thermal reactors because of their high absorption coefficient. Zirconium is manufactured by the Kroll process or, in purer form, by the van Arkel process. Annealing must be carried out *in vacuo* if the formation of an oxide coating is to be avoided. Pure zirconium has a tensile strength of no more than 16 tons per square inch but zirconium alloys are being developed which have similar properties to those of titanium alloys at 500°C. Aluminium, tin and titanium are suitable alloying elements. Zirconium would be a suitable construction material for atomic energy plant but for its hafnium content, which results in a very high absorption coefficient for thermal neutrons. Tantalum is highly corrosion-resistant and has certain gettering properties with respect to hydrogen, nitrogen and oxygen. Annealing of tantalum must be carried out in vacuum in order to prevent picking up oxygen which makes the metal brittle. Tantalum has a high absorption coefficient for slow neutrons and is therefore unlikely to be used in reactors themselves. Niobium has properties similar to those of tantalum but is not yet available in commercial quantities. Molybdenum is useful to the chemical engineer because of its high temperature resistance and high thermal conductivity, but is limited in its application by its low resistance to oxidation, and poor welding quality. In conclusion the article reports on recent progress in fabrication techniques.

Sommaire: Les méthodes employées dans la production d'un certain nombre de nouveaux métaux déjà utilisés ou allant être utilisés dans la construction d'équipements pour l'énergie atomique, sont décrites, ainsi que les proprietés de ces nouveaux métaux.

Paper by L. Rotherham *Chem. & Industr.* 18.9.1954 1164-1170

The Basic Factors Governing the Degassing of Metals in the Solid State 216/I

Germany. Gas contained in metals is known to have an adverse effect on the properties of a metal. Gas may be present in the solid metal either as (1) gas adsorbed by the metal surface, or (2) gas dissolved in the body of the metal or (3) free gas held in pockets within the metal. Gases are adsorbed by the surface of metals in one of two ways, (*a*) by physical adsorption or (*b*) by chemical or activated adsorption. The former applies if the gas and the metal do not interact chemically. In the immediate vicinity of a surface certain forces are acting, partly of a purely electrical nature (repulsive), and partly resulting from van der Waals forces and/or chemical valency forces (attractive). At a short distance from the surface the attracting forces are predominant and the molecule approaching the surface is subjected to an increasing force up to a point where it reaches a maximum and forms a potential well. Depending on the type of adsorption this well is either shallow or deep and the forces in action cause the molecule to be retained in the zone of the boundary layer as long as its own energy is not large enough to permit it to detach itself. The retention of the gas molecules in the adsorbed layer depends on the pressure and temperature conditions. The adsorbed layer is in equilibrium with the surrounding gas if the number of molecules arriving and departing from the boundary layer is equal. The author proceeds to discuss adsorption under isothermal and isobaric conditions. In the latter case, as the temperature rises, the amount of gas collecting in the adsorbed layer increases until it reaches a maximum at 300°C when it decreases again. At this stage activated adsorption is initiated if conditions permit. In the case of activated adsorption, gas atoms may enter the metal lattice and form a solution. There are two types of solution: (*a*) The gas combines with the metal by substitution, forming hydrides or oxides, for instance. (*b*) Atoms of the gas penetrate into the interstices of the metal lattice (the case of a 'simple' solution). The formation of a simple solution is an exothermic process whereas (*a*) is endothermic. The solubility of a gas in a metal also depends on the pressure. The pressure dependence is expressed by

$$c_2 = \frac{1}{K \cdot \sqrt{p_1}}$$

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where c_2 is the mole-concentration of the gas dissolved in the metal, p_1 the pressure of the gas in the molecular state surrounding the metal and K a constant. Examples are shown of simple solutions and compound solutions for various metals. Finally the diffusion of gas through metal is discussed. The diffusion mechanism is not yet fully explored but it is clear that it is temperature-dependent and formulae expressing the temperature dependence are given. The author concludes that the application of vacuum offers the best means for degassing metals, as all three phenomena, *i.e.* adsorption, solubility and diffusion are pressure dependent. The application of heat, however, has to be carried out with some care as there is an optimum range of temperatures in each case, determined by a number of extraneous factors. The time required for the proper degassing of metals may vary appreciably, *e.g.* diffusion processes depend on the concentration gradient and normally take a long time. It is comparatively easy to remove gases adsorbed on the surface but often very difficult to remove gases from the interior of the material. Certain nitrides and oxides are highly temperature-resistant and can only be eliminated when melting temperature has been reached.

Article by W. Scheibe *Vakuum-Tech.* 3, Sept. 1954 67-71

Sommaire: Revues des connaissances actuelles sur les mécanismes qui facilitent l'introduction de gaz dans des métaux à l'état solide, afin d'étudier le dégazage des métaux.

Some Observations Relating to the 'Wetting' of Titanium with Mercury 217/I

See Abstract No.: 265/I

The Mechanism of Drying of Solids. Part V. Freeze Drying of Clay and Vacuum Drying of Wet Clay 218/I

United Kingdom. As the drying of frozen clay proceeds, the ice surface in the material recedes continuously and the vapour arising from the ice surface has to pass through a layer of dried material which thickens continuously. The rate at which the vapours pass through the dried layer, *i.e.* the drying rate, is governed by the size and length of the pores formed in that layer. In the case of clay the pores are relatively large and for that reason Poiseuille flow can be assumed. Thus, the mass flow rate is proportional to the square of the pressure and can be expressed by

$$\text{Mass flux } G = k \frac{P^2}{L}$$

where L is the thickness of the dried layer, P the upstream or vapour pressure of the ice, k a constant equal to $1/\rho$, ρ being the specific resistance to flow. In order to confirm that Poiseuille flow conditions apply in the case of clay, preliminary experiments were conducted by preparing a paste from 40 parts by weight of water for every 100 parts of clay, forming a disc of that paste and placing it over the mouth of an evaporator pot which held a sheet of ice at the bottom. The heat input was adjusted so that the vapour flux arising from the ice surface was the same all the time. Measurements were taken for various water vapour pressures and are given in a table, partly reproduced below.

Experimental Determination of the Flux

G' $\frac{g.}{cm.^2 \times hour}$	L $mm.$	P $mm.\ Hg$	ρ' $\frac{cm. \times hour \times (mm.Hg)^2}{g.}$
0.49	2	7.3	54
0.32	2	5.9	55
0.073	5	4.6	57

Having established in these experiments that Poiseuille's law applies, a fresh series of experiments was conducted to determine the drying rate of a bed of frozen clay. The apparatus employed is described in *Trans. I. Chem. Eng.* **32**, 1954, No. 3, 192. Heat, applied to the base of the frozen clay layer, was kept constant throughout the experiment. After initial oscillations the mass flux fell to values as low as 0.05 in each run. Details of the results are given in the form of graphs and prove that the drying rate is inversely proportional to the thickness of the layer of the processed material. In these experiments the specific resistance was found to be approximately 100 as compared with **57** in the preliminary experiments. This discrepancy cannot be explained as no shrinking occurs during the drying of frozen clay. By a similar method the evaporation of water from wet clay was investigated. A 40–100 paste was used and the rate of evaporation determined at constant heat input. Three distinct phases were observed: While the moisture content of the clay was reduced from 40% to 21%, the water evaporated at the surface and the drying rate was constant. As the moisture content was reduced from 21% to 6%, a wet-dry interface moved upwards through the clay layer and, finally, drying from 6% to 0% involved the removal of adsorbed water. In both the latter phases the drying rate was falling off, most steeply in the final phase. The paper concludes with a brief report on experiments carried out on the adsorption of water vapour on clay. The method employed in the investigation permitted the preparation of a series of isotherms shown in graphs where the water vapour pressure is plotted against weight of water adsorbed on a given quantity of dry clay. The shape of the curves indicates that down to about 15% moisture content free water is present in the clay, whereas lower moisture content represents water in the adsorbed condition. As a further step isosteres were constructed on the base of the isotherms which facilitated

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calculation of the heats of adsorption. The heat of adsorption for 8% water content was found to be 11,800 cal./g.mole which compared with a latent heat of evaporation of pure water = 11,000 cal./g.mole. These figures permit the conclusion that at 8% water content or less the moisture present consists essentially of adsorbed water.

Sommaire: On donne des détails et des résultats sur des expériences faites afin de déterminer les facteurs principaux qui gouvernent le séchage d'un matériau poreux et humide, soit par rafroidissement, soit par le vide.

Article by R. F. Strickland-Constable & E. W. Bruce *Trans. I. Chem.E.* **32**, No. 3, 1954 199-203

The Effects on Biological Materials of Freezing and Drying by Vacuum Sublimation. I. Development and Testing of Apparatus 219/I

See Abstract No.: 73/IV

The Effects on Biological Materials of Freezing and Drying by Vacuum Sublimation. II. Effect on Influenza Virus 220/I

See Abstract No.: 74/IV

Gas and Water Vapour Absorption of High Boiling Liquids at Pressures Below 10 mm. Hg. 221/I

See Abstract No.: 189/III

Apparatus for Molecular Distillation 222/I

France. It is known that mixed liquids can be separated by vacuum distillation procedures. However the vacuum employed in the column still is normally of the order of a few millimetres Hg and in many instances the boiling point of the material to be processed is still too high in that pressure range to eliminate all risks of deterioration by carburisation or decomposition generally. In the manufacture of perfumes and cosmetics it is necessary to fractionate essential oils or to prepare monogylcerides of high concentration. These processes require a vacuum as high as 10^{-5} mm. Hg. The use of the molecular distillation method has proved particularly beneficial in that field. The main criteria determining the choice of the distillation method to be employed are the molecular weight of the substance and the highest permissible boiling point. The author proceeds to describe molecular distillation apparatus of a particular design where the material to be treated flows down the walls of a vertical cylinder in the form of a film less than 1 mm. thick. The apparatus is composed of a number of identical units, each of which yields one fraction, the whole plant being assembled from as many units as fractions are required, not counting the residue. One model of the apparatus described facilitates continuous fractionation at a throughput of 500 to 1,500 g. of processed material per hour. It permits the treatment of mixed liquids with up to 5 constituents. A second model consists of a single unit only and is included to serve as a pilot-plant.

Sommaire: Une brève revue sue les qualités des méthodes de distillation moléculaire employées dans l'industrie des parfums, et détails sur des colonnes de distillation spécialement développées pour ce travail.

Article by R. Fouche *Ind. Parfum.* **9**, August 1954 275-276

The Organisation and Purpose of the Liquid Helium Pool 223/I

See Abstract No.: 69/IV

New Developments in Vacuum Testing 224/I

United States. The uses of vacuum are discussed as applied to testing procedures for a variety of purposes in industrial production. Details are given of a so-called 'Bubbler' tester which gives a visual indication of the presence or absence of a leak in a component. It essentially consists of a cell, holding a small amount of fluid, with a clear glass plate in front and a diffused glass plate at the back, the latter being illuminated from behind. The cell is inserted in a vacuum system. For the purpose of the test the component is connected to the vacuum system and the system is evacuated. As the air is removed from the component, bubbles form in the fluid and are clearly visible against the diffused background of the cell. If the formation of bubbles ceases after an initial period the component is leak-tight. If the formation of bubbles continues, the component has a leak. The method is rapid and eliminates the necessity of immersing the test piece in a fluid. Reference is made to other vacuum testing methods such as the use of vacuum gauges for measuring the angle of engine valve seats. Finally there is a brief description of the use of vacuum for the determination of the size of holes as small as 0.0065 inch in diameter with a ± 0.0001 inch tolerance, by applying the principle of flow rate measurement.

Sommaire:Description d'un certain nombre de méthodes d'essais par le vide employées dans l'Industrie; des références particulières sont données sur leur emploi dans l'Industrie Automobile.

Article by L. Sterne *Autom. Industr.* **72**, 1.8.1954 106

Removing Carbon Bisulphide from Viscose Fibre with a Liquid Seal Vacuum Pump 225/I

See Abstract No.: 196/III

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13 — ELECTRICAL SCIENCES — 13

Measurement of the Electrical Resistance of Metal and Alloys at High Temperatures 226/I

See Abstract No. : 259/I

Phenomena Associated with the Disintegration of Fuse Wires 227/I

United Kingdom. The disintegration of fuse wires of approximately 0.92 mm. diameter under short-circuit conditions has been studied at atmospheric and reduced pressures. In atmosphere, current slightly in excess of the limiting current produces a temperature peak near the centre of the fuse wire, eventually causing the wire to break in that area. A substantial excess current on the other hand will produce a uniform temperature rise over the whole length of the wire. The present experiments have shown that as the temperature approaches the melting point, the wire is twisted into a shape approximating to an unduloid and, as the portions of smaller cross-section tear, a number of spherical globules are formed in succession during an interval of a few micro-seconds. The flow of the current across the gaps between the globules is maintained by a series of small 'elementary arcs'. As the globules evaporate a single arc forms reaching from one terminal to the other. In order to secure proper action of the fuse the arc must be quenched, *i.e.* the voltage required to maintain the arc must be larger than the circuit voltage. Typical of fuse wire disintegration at atmospheric pressure is a sudden transient voltage rise at the moment when the wire breaks up. Experiments at low pressure were carried out in commercial vessels modified to facilitate evacuation to 10^{-3} to 10^{-4} mm. Hg. In a vacuum, the transient voltage was greatly reduced. This is explained as follows: The first elementary arc serves as a source of ions which, due to the long mean free path, rapidly reach all parts of the vessel and effect the breakdown of the metallic vapours present. Thus a discharge is established by-passing all remaining solid portions of the wire. The elementary arc in atmosphere generates ions just the same but due to the absence of a rarefied atmosphere the discharge is confined to the gap where the arc occurs. In atmosphere, break-up of the wire is indicated by a loud detonation resulting from the sudden expansion of the air and metallic vapour but no audible signs have been noticed during disintegration under vacuum. The pressure-wave effect just mentioned is made use of in the so-called expulsion fuse, where the sudden expansion of the air in the vessel has a blow-out effect. At sub-atmospheric pressures the arc requires a smaller voltage for its operation. A means of improving the performance of a low-pressure a.c. fuse, for instance, is to prevent restriking of the arc. In experiments with fuse vessels some 35 cm. long, restriking occurred irrespectively of the material of the fuse wire at voltages exceeding 1,000, but at 350 V the nature of the wire material had a decisive effect. Tungsten, for example, has a high evapora-

Restriking Characteristics of Elements at Reduced Pressure with Copper Electrodes

Metal	*Vaporisation temperature deg. Cent.*	*Tendency to restrike at 350 V r.m.s.*
Zinc	887	At each current zero
Silver	1950	At first or second current zero
Copper	2300	No restriking
Tungsten	5900	No restriking

tion temperature and consequently a high rate of condensation. Thus, the pressure conditions which exist prior to the disintegration of the wire are restored almost at once. Improved low-pressure fuse action can be obtained by covering the electrodes with insulating and heat-resisting material. This reduces electron emission from the cathode resulting in a smaller arc current. A device designed on these lines is described and works satisfactorily at about 500 V.

Sommaire: Le mécanisme de la désintégration de fils fusibles aux pressions atmosphèrique et sub-atmosphèrique a été étudié et des recommendations sont faites pour la construction de fusibles limitant un courant alternatif sous vide pour des tensions de 500 V.

Article by A. E. Barrington *Engineer* **198**, 6.8.1954 187-189

Gas Discharge Lamps 228/I

Holland. Production and performance data of the various types of gas discharge lamps available today are given. There are three major types of such lamps: (*a*) Low-pressure mercury lamps, (*b*) high-pressure mercury lamps and (*c*) special discharge lamps employing other vapours or gases. The lamps of type (*a*) are tubular in shape throughout, varying in diameter from 24 to 38 mm. and reaching a length of 120 cm. Their consumption ranges from 20 to 65 watt for supplies of about 220 V. The lamps have a fluorescent coating which is produced by applying a nitro-cellulose suspension of the fluorescent powder to the inner walls of the tube prior to sealing, and subsequently eliminating the nitro-cellulose by heat treatment. Then the tungsten filament electrodes, coated with an alkaline earth carbonate, are sealed into the ends of the tube, and the tube is placed in an oven and evacuated to approximately 1 mm. Hg. While pumping continues, the electrodes are connected to a supply and the carbonates converted into oxides. When all the carbon dioxide liberated in the process has been pumped away, a drop of mercury is introduced into the tube, *via* the pumping stem, which evaporates as it hits the bottom of the tube. The rising mercury vapours assist in displacing the residual gases in the tube at that stage. During the cooling down of the tube the discharge is struck, an additional predetermined quantity of mercury is

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introduced into the tube, and argon is admitted at a pressure of a few mm. Hg. The lamp is sealed off and run under normal operational conditions to effect final clean-up. The light emission of the fluorescent coating is caused by the energy radiated from the ionised mercury vapour at the resonance line 2,537Å. Lamps to be used for sterilisation purposes have no fluorescent coating and are made from glass which transmits the energy radiated at 2,537Å. The operational pressure of the mercury vapour in type (*a*) lamps must not exceed 5×10^{-3} mm. Hg as otherwise self-adsorption takes place weakening the emission at the resonance line. The presence of a rare gas which has a higher level of excitation does not affect these conditions. The argon in the lamp has two functions: Firstly it acts as the carrier gas and initiates the discharge, as the vapour pressure of mercury at room temperature is not high enough. Secondly, the presence of the carrier gas reduces the mean free path of the electrons and thus assists ionisation generally. Excitation of the vapour atoms depends on the current density in the discharge. The length and diameter of the tubes are governed by the requirements of low current density in the discharge to obtain maximum radiation, and a large voltage drop along the discharge column. In practice, the small pressure of the vapour results in a low voltage gradient. For this reason the tubes are made as long as possible. Stability of operation is ensured at a voltage drop of 100 in the discharge column of lamps operating on a 220 V supply. This necessitates a length of about 1 m. at the normal tube diameters of 25 and 40 mm. The lamps of type (*b*) have a higher light output than those of type (*a*). This is achieved by operating the lamp at mercury vapour pressures from 1 mm. Hg upwards. As a result the wall temperature of the lamp rises and special glasses, even quartz glass, are required to ensure a reasonably long service life. Various design features of the high-pressure mercury lamp are discussed in detail. The spectrum of the high-pressure lamp has lines in the violet, blue, green and yellow range and in addition shows a weak continuous band but the intensity of the latter is too small to counteract discoloration of the objects illuminated. For that reason, many high-pressure lamps have a fluorescent coating. Reference is made in this connection to the combined high-pressure mercury and tungsten filament lamp. Certain types of high pressure mercury lamps designed for other than pure illumination purposes are made in the shape of a bulb which facilitates the production of high current densities. High current densities have also been obtained in tubular types of lamps by reducing the tube diameter to 1–2 mm. but such lamps require special cooling arrangements and this led to the development of the water- or compressed-air-cooled high pressure mercury lamp. Finally, the author discusses lamps representative of type (*c*) such as the sodium vapour lamp.

Sommaire: Discussion sur les détails de construction et d'exploitation de lampes à mercure à basse pression, à haute pression, et de certaines lampes employant des vapeurs métalliques spéciales.

Article by W. Elenbaas *Vakuum-Tech.* 3, July 1954 36-42

Bulk Vacuum System

229/I

United States. In order to sensitise screens for t.v. colour tubes the screens are placed in frames and vacuum is applied to ensure that the screens are kept in a flat and wrinkle-free position during treatment. In the past each frame formed a separate unit. The present note describes briefly a 'vacuum storage system' where ten such frames are combined in one single unit and are served jointly by one vacuum pump aggregate. An 850 gal. manifold is inserted in the system between the pump aggregate and the frames. The pump aggregate consists of 6 pumps but where variations in load make it unnecessary to use all pumps, one or more of the pumps are cut out. The equipment described reduces operating cost as well as ensuring permanent and adequate pumping capacity. The principle is recommended for use in other large-scale vacuum operations.

Sommaire: Brève description d'un appareil à vide pour production, employé dans le traitement d'activation des écrans de tubes de Télé-Vision en couleur.

Note by Anon. *Compr. Air. Mag.* July 1954

Printed Circuit Technique

230/I

Germany. Printed circuits are employed in telecommunication equipment and high frequency technique, and consist of conducting layers, in the majority of cases of a metallic nature, applied to non-conducting materials, to provide current conductors, capacitances or resistances. The methods employed for the application of the metallic layers are spraying, casting, painting, chemical deposition and more recently high vacuum evaporation. Various methods based on these techniques are described. A recently developed non-vacuum metallisation process, particularly suitable for the production of conductors on plastic materials, provides for a copper foil to be pressed on to the plastic face in the hot state. Subsequently, the outlines of the conducting strips are marked on the copper foil and coated with an acid-resistant lacquer. Finally all parts of the foil not covered by the lacquer are removed by etching with an acid. Where ceramics are the base material, firing-on of silver or other precious metals is still popular. Much attention has been paid to the development of suitable silver preparations. New fluid silver preparations are available which can be applied by gun spraying, employing masks to cover the parts which are not to be silvered. An alternative method is the silk screen process, details of which are given. After application, the silver layers are fired at a controlled temperature varying from 600 to 700°C to avoid excessive evaporation. Silver layers of the kind described can be soft soldered and re-inforced by anodising, if required. Heat sensitive materials such as bakelite, thermo-plastics or paper can be coated with special silver preparations substituting air-drying of the silver coat for the firing process. The maximum heat required in this method is about 100°C. For the printing of resistances a suspension of colloidal graphite in a silicone solution is employed. The finished printed circuit requires a coating of lacquer to give protection against moisture and mechanical damage. The composition of the lacquer depends on the nature of the base and the properties of the metals deposited. Normally, silicone solutions in toluol, or polystyrol lacquers are used. In recent years vacuum techniques have been employed for the production of printed circuits. Sputtering is essentially confined to the production of conducting layers from gold or platinum, also the production of semiconductors from metal oxides, but vacuum evaporation has a less restricted application. Layers of copper, zinc, tin, aluminium and silver can be obtained on any type of base such as ceramics, piezo-electric crystals,

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glasses or thermo-plastic materials. The metallic layers can be produced in appreciable thicknesses, and are uniform and show good adhesion properties. If the necessary care is taken, soft soldering is possible. Finally, in a separate processing step, water-repellent oxide layers, which are at the same time abrasion-resistant, can be evaporated on top of the conducting layer, serving as a protective coat. Vacuum coating plant designed for the purpose provides for continuous operation and is normally fitted with the coating chamber in a horizontal position to facilitate loading and unloading.

Sommaire: Discussion sur toutes les méthodes employées à prèsent pour la production de circuits imprimés, avec référence sur les méthodes d'évaporation sous vide et leur avenir dans ce domaine.

Article by E. Dürrwächter *Elektrotech. Z.* **6**, March 1954 73-75

The Zinc Coating of Paper for Capacitors by Vacuum Evaporation 231/I

See Abstract No. : 164/III

15 — PHYSICS — 15

Two-Layer Anti-Reflection Coatings for Glass in the Near Infra-Red 232/I

See Abstract No.: 157/III

On Polarisation by Transmission with Particular Reference to Selenium Films in the Infra-Red 233/I

United Kingdom. The method of polarisation by transmission through a pile of plates or films inclined to the incident radiation at Brewster's angle is employed for the determination of optical properties of metals and semiconductors in the infra-red and ultra-violet region. In this method polarisation is estimated by the formula of Provostaye and Desains which is based on the assumption that there are multiple reflections among all the surfaces. However it has been noticed that practical observations do not coincide with the theoretical estimates based on this formula. In their investigations the authors employed evaporated selenium films in the infra-red and cast zapon lacquer films in the visible region. In the case of a polarising element consisting of eight selenium films, each 3 μ thick, the observed value exceeded 99% and the calculated value was 89%. The authors investigated the performance of these polarising elements and report the following findings: Absorption may be neglected as long as the films are of small thickness. Interference among films can be ignored but interference within the individual film has to be taken into account. The total lateral displacement of the beam caused by the reflection of the eight selenium films in the polarising element is of the order of 14 μ. At this order of magnitude phase differences can be regarded as randomly distributed and therefore there is little regular interference, and the phase change introduced by such components is negligible as long as the refractive index is high. Multiple reflection among films, assumed to be present in the Provostaye and Desains expression, is negligible due to the combined effect of the intervals between the plates ($\frac{1}{4}$–inch) and the aperture dimensions ($\frac{3}{4}$–inch diameter) which limit the number of displaced beams. Making due allowance for the conditions as established in this investigation good agreement is obtained between the calculated and observed values.

Sommaire: On a étudié la méthode établie pour l'estimation de la polarisation par transmission au travers d'une pile de plaques avec films, inclinée au rayon incident, et des différences trouvées dans ces observations ont été évaluées.

Article by G. K. T. Conn & G. K. Eaton *J. Opt. Soc. Amer.* **44**, July 1954 553-557

Usefulness of the Polarimetric Method for the Determination of the Refractive Index and of the Thickness of One Thin Film on Glass or on Metal 234/I

Czechoslovakia. The author refers to the polarimetric method for determining the refractive index and thickness of a thin film on glass described by him previously (*J. Opt. Soc. Amer.* **37**, 1947, 145). Doubts have been expressed whether this method can be applied to films of a thickness of less than a few hundred angstrom. The author points out that the merits of the method have been confirmed in the meantime for films thicknesses down to 10 Å. The determination of the refractive index of aluminium oxide films produced by anodising aluminium mirrors has proved difficult at thicknesses below 500 Å solely for the reason that the relationship between the thickness and the measured ellipticity angle is very much the same for different values of the refractive index of the film. But if the refractive index is known, determination of the thickness of the film presents no difficulty.

Sommaire: On discute l'étendue et les limites d'utilisation de la méthode polarimetrique dans la détermination de l'indice de réfraction et de l'épaisseur de couches minces, déposées sur verre.

Letter by A. Vasicek *J. Opt. Soc. Amer.* **44**, August 1954 664-665

New Types of Interference Filters of Greater Areas and Higher Contrast 235/I

Denmark. Narrow pass-band optical filters of the Fabry-Perot type had been produced in a previous investigation using two silver layers and a dielectric layer of cryolite. This method has been extended to the production of filters of higher contrast involving four metallic layers and three dielectric layers. An evaporation plant was specially built to prepare filters of up to 25 cm. diameter. The steel vacuum chamber was 60 cm. in diameter and 80 cm. high, and was evacuated by a 9 inch. silicone diffusion pump. The thickness of the 25 cm. diameter filters was uniform within 0.5%. Film thickness was measured polarimetrically through side-ports in the bell

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jar. Cryolite was chosen for the dielectric layer in preference to magnesium fluoride because it presents less difficulty when evaporating large quantities. A stable filter could only be obtained if the cyrolite was evaporated at a very slow rate. The thickness of the silver layers was determined by means of transmission measurements on test plates. Several filters of this type were produced. It was observed that in order to obtain an adequate band width the inner metal films had to be made thicker than the end films. This agreed with the calculations. Filters of 20 sq. cm. were produced with a peak transmission as high as 0.35–0.40. At a wavelength of 500 Å from the peak (second-order films) the transmission was less than 0.001.

Sommaire: Description de la méthode de production et des performances de grand filtres optiques Fabry-Perot du type à bande passante étroite.

Article by A. Hermansen *Nature* **174**, 31.7.1954 218-220

Cathodoluminescence of Air, Quartz and Metal Films 236/I

South Africa. It has been reported that thin metal and other films when exposed to α-particle irradiation show light emission. It is known that air and fused quartz in these conditions show a cathodoluminescence spectrum which in the case of the former consists of a series of lines in the visible and near ultra-violet, and in the case of the latter of a continuum extending over the same spectral region. In order to obtain confirmation of luminescence effects of thin metal films under these conditions the authors conducted experiments with thin foils of gold, silver, aluminium and tin. A quartz spectrograph was employed in the experiments, and electrons of an energy of 100 keV were extracted from a discharge tube through a metal Lenard window on to the specimen which was placed in external contact with that window. The method described has the advantage of producing potentially higher luminescence and eliminating the self-luminosity of the α-particle source. No luminescence effects were obtained from the specimens in these experiments. When the foil was mounted on a quartz plate the spectrum observed was identical with that for quartz alone, except for a reduced intensity due to dissipation of the radiated energy in the specimen. Similarly the air spectrum only was obtained when the specimen was mounted in air.

Sommaire: Des expériences ont été faites pour confirmer, une émission de lumière provenant de certaines feuilles de metal soumises à un bombardement électronique; les résultats sont négatifs.

Letter by J. B. Birks & W. A. Little *Nature* **174**, 10.7.1954 82-83

Perfection of Contour by Vacuum Deposited Thin Films 237/I

United Kingdom. The fidelity of contouring of surface micro-steps by evaporated thin deposits of silver, cryolite and zinc sulphide is studied by precision multiple-beam interferometry. As a known step, a spiral on silicon carbide is used and successive layers of thin film are deposited on this, the step being re-measured for each increment of deposit. With silver the contour is exact to within plus or minus 3 Å up to an overlying silver thickness of some 27,000 Å, although the step height being examined is only 480 Å in height. Up to thicknesses of 6,000 Å the measured step is correct very close to the step edge but beyond this thickness a defect sets in, such that there is a build-up on the step riser which reaches a maximum of about 100 Å close to the edge. With cryolite and zinc sulphide these defects are notably more marked. The experiments establish that the type of silver coating used in interferometry can be considered to contour surfaces perfectly. Multilayers may tend to obliterate micro-structure if this is on a very small scale in extension. (*Author*)

Sommaire: La fidélité d'épouser des denivellations de surface par évaporation de dépôts d'argent minces, de cryolithe et de sulfure de zinc est étudié, avec un interferomètre à faisceau multiple de précision.

Article by S. Tolansky *Vacuum* **4**, Oct. 1954 456-463

Thickness Measurements of Thin Films by Multiple Beam Interferometry 238/I

See Abstract No.: 154/III

Light Absorption and Electrical Properties of CdO Layers with an Imperfect Lattice 239/I

See Abstract No.: 167/III

An Electron-Diffraction Examination of Thin Films of Lithium Fluoride and Copper Prepared by Vacuum Evaporation 240/I

See Abstract No.: 159/III

Structure and Texture of Evaporated Nickel Films Determined by the Electron Microscope and Electron Diffraction 241/I

Holland. In order to study properly the processes occurring at the surface of a catalyst, it is imperative to have available a surface of known structure and texture. The authors report on an investigation of nickel film surfaces, the films having been prepared by an evaporation method which excluded the presence of organic vapours. The equipment was made of Pyrex glass and was baked for several days at 150°C preliminary to the evaporation process. As a result a pressure of 10^{-7} mm. Hg could be maintained. The nickel was slowly evaporated at 1,240°C at a rate of 0.005 to 0.15 milligram per hour and deposited on a glass substrate. The temperature of the substrate was kept at a pre-determined temperature by means of a constant temperature

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bath. The amount of nickel contained in each deposit was not assessed by thickness measurement but in terms of number of atoms per square centimetre. (A nickel film of 1,000 Å thickness contains about 9×10^{17} atoms per sq. cm. if the density of the material is assumed to be that of the bulk material, *i.e.* 8.8). For examination in the electron microscope, carbon replicas of the films were prepared and shadowed with palladium. The magnification employed was about 20,000. Reference is made to experiments published by Beeck (*Phys. Rev.* **55**, 1939, 601), who stated that in the presence of an inert gas only slight orientation could be observed. The authors' experiments show that, irrespective of the presence of any inert gas, good orientation is obtained with (110) faces parallel to the horizontal. The degree of orientation depends on the condensation temperature and on the thickness of the layer. The speed of evaporation has no influence. At very low temperatures (liquid nitrogen) no orientation takes place. This is explained by the fact that the atoms reaching the substrate must be mobile in order to obtain a well-defined orientation. Non-orientated surfaces showed a predominant number of crystal faces in the horizontal plane but no horizontal planes of crystals were noticeable at the top of orientated surfaces and it must be concluded that (110) face crystals with planes in the horizontal are not responsible for any increased catalytical activity which may have been observed. The observations made in these experiments are interpreted on the base of the theories developed by Stranski *et al.* for the kinetics of homo-polar crystal formation and permit the deduction that the crystals in the top face of the layer are tetradecahedral. In conclusion it is stated that the direction of the orientation of the crystals in a metal film depends entirely on the crystalline nature of the material in question. A material consisting of face-centred cubic crystals will crystallise around the (110) orientation axis, and body-centred cubic crystals will orientate around the (111) axis.

Sommaire: La structure de couches de nickel pur évaporé a été étudiée, afin de trouver les facteurs de base gouvernant la catalyse hétérogène.

Article by W. M. H. Sachtler G. Dorgelo & W. van der Knaap *J. Chim. Phys.* **51**, Sept. 1954 491-496

A High-Temperature Microwave Spectrometer

242/I

United States. A spectrometer for the measurement of microwave absorption by a gas yields rotational spectra which give information on molecular and nuclear structure. Materials suitable for examination must consist of diatomic molecules which have a molecular electric dipole moment and a rotational constant not exceeding 100,000 Mc (the limit of the instrument). So far only room temperature microwave spectrometers have been employed, which restricts the materials suitable for examinations still further to those with a vapour pressure exceeding 10^{-2} mm. Hg at room temperature. In the present article details are given of the design and operation of a high temperature instrument of this kind where the absorption cell can be raised to a temperature of 1,000°C, extending the application of the instrument to the examination of several hundred different materials. The present instrument is of the square-wave Stark-modulated type. Stark-modulation is produced by introducing a long conducting septum parallel to, and electrically insulated from, the broad face of the wave-guide cell. The voltage, zero during half of each cycle, applied to this septum, modulates the resonant absorption frequencies of the gas or vapour in the cell. High temperature operation of the instrument requires radical alteration of the conventional design to overcome failure of the construction materials ordinarily used for the purpose. The Stark septum in the new design consists of gold-plated nickel, supported by a few small square prismatic pieces of Forsterite in a position 0.085 inch. away from the wave-guide holes, and the cell is mounted in a vertical position to avoid sagging between the points of support. The cell is of the same material and 5 ft. long. Thus the whole of this assembly consists of highly corrosion-resistant material. The cell is situated in a vacuum chamber pumped down by an oil diffusion pump unit of a speed of 275 l./sec. to 10^{-4} mm. Hg. This prevents oxidation of the outside of the wave-guide cell and simplifies window-assembly construction. The window must be transparent to microwaves, withstand the pressure difference between the space outside and inside the cell, tolerate high temperatures and finally must be thin enough not to cause appreciable frequency-sensitive reflections. In the new instrument there are two windows, one in the high-temperature region made from 0.010 inch fused silica, the gasket employed being corrugated steel shim, and the other in the cool region designed as a conventional mica window. The latter has to withstand a pressure difference of 1 atmosphere but the former is virtually relieved of all strain on this account. Tests with NaCl vapour in the cell, heated to 800°C, with a vapour pressure of 10^{-2} mm. Hg showed that gas leaks across the window at a rate of 3 mg. per hour, *i.e.* half the initial gas content of the cell leaks into the vacuum chamber in about 0.8 seconds. This condition has a favourable effect in that the leakage assists in evacuating the wave-guide eliminating any impurities present in the cell due to outgassing, etc. The instrument has been employed with success for the observation of the spectral lines of materials such as KCl, NaCl and CaCl.

Sommaire: On donne des détails sur un spectromètre à micro-onde récemment développé qui permet de chauffer la cellule d'absorption jusqu'à environ 1,000°C.

Article by M. L. Stitch A. Honig & C. H. Townes *Rev. Sci. Instrum.* **25**, Aug. 1954 759-764

The Friction of Non-Metallic Solids

243/I

United Kingdom. In metallic friction the total area of contact between adjacent surfaces is a minute fraction of the surface areas involved. Plastic deformation increases this area proportionally to the load applied and, because of the high loading at these points, adhesion and shearing gives rise to the observed frictional forces. Oxide and other surface films reduce the extent of metallic contact. If metal surfaces are placed together in a vacuum and these surface films are removed, gross seizure occurs and sliding is impossible. The friction falls again when air is admitted. Very small displacements, before sliding commences, are irreversible and the electrical contact resistance falls, indicating that plastic rather than elastic deformations have occurred. The resistance drop is much greater for metals with oxide films than for oxide-free surfaces. Much use is made in this work of reflection electron microscopy employing an electron beam at grazing incidence on the surface to

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be examined. This eliminates the need for replicas, a constant potential source of inaccuracy. For plastics the mechanism of friction is essentially the same as for metals, but under light load some elastic deformation of the contact regions occurs and the coefficient of friction increases as the load falls. Polytetrafluorethylene (P.T.F.E.) has a particularly low coefficient of friction (μ=0.05–0.1), changing little under vacuum. If this material is incorporated in sintered copper, a solid having high thermal conductivity and low friction results.

Coefficient of Friction of Metal Impregnated with P.T.F.E.

Temperature °C	*Copper*	*Impregnated Copper*
15	about 1.0	0.05
100	about 1.0	0.05
200	about 1.0	0.05
250	greater than 1	0.05

Brittle solids, such as rock salt, exhibit plastic flow under the high loadings found at points of contact, and have an enhanced shear strength which, for rock salt, is ten times the bulk shear strength. Gross seizure observed under vacuum with metals does not occur. The behaviour of elastic solids such as rubber is similar to that of plastics at light loading, the coefficient of friction being proportional to the 2/3 power of the load. Diamond has been found to belong to this group. Under vacuum the friction between diamond surfaces rises—but not to the same extent as with metals. Recently, using reflection electron microscopy, some evidence of plastic deformation during the polishing of diamonds has been obtained. The coefficients of friction parallel and perpendicular to the axis on the (110) face have been found to be μ=0.5 and μ=0.04 respectively. Ice has a low friction only near its melting point, where pressure-melting is a factor. At low temperatures, the static friction of ice is high. The apparently low frictions encountered during ski-ing are caused by frictional heating of the sliding surfaces, melting the snow.

Influence of Speed on Friction: On Ice at –10°C

Ski Surface	*Coefficient of Friction, μ*		
	Static	*at 3 cm./sec.*	*at 5 m./sec.*
Waxed wood	0.20	0.18	*c.* 0.02
Lacquered wood	0.40	0.36	*c.* 0.03
Perspex	0.35	0.32	*c.* 0.03
Aluminium	0.38	0.34	*c.* 0.04

In the solid state, the inert gases might be expected to have low frictions as they are held together by the comparatively weak Van der Waal forces. However, the static friction of solid krypton is high (μ=0.8) and, like ice, falls only near its melting and boiling points (which are close together). Solid carbon dioxide behaves similarly. The friction between molecularly flat surfaces prepared from cleaned mica sheets is high, the surface damage being akin to that produced by shearing solid mica.

Article by F. P. Bowden *J. Inst. Petrol.* **40**, April 1954 89-101

Sommaire: Revue générale sur les connaissances actuelles des phénomènes de friction entre solides métalliques, solides non-métalliques, minèraux et gaz solidifiés.

Importance of Thermal Equilibrium in Measurements on Powdered Insulators in Vacuo

244/I

United States. Experiments are reported which demonstrate that the true temperature of powder used as a thermal insulator in vacuum is determined by the radiation energy transmitted to and from the powder unless good contact is ensured between the powder and the surface of the container, maintained at the desired temperature. The experiments were carried out with specimens of self-activated zinc sulphide phosphor, excited with 3,650 Å radiation, held in a small copper dish, 50 mils thick. The dish was placed on the nickel-plated copper plate forming the bottom of a container which could be exhausted and cooled to liquid nitrogen temperature, and which had two windows, one for observation and one for admission of the exciting radiation. A small heater was provided to heat the nickel-plated copper plate to 200°C. Experiments in the pressure range from atmosphere down to 10^{-1} mm. Hg showed that the temperature of the phosphor layer exposed to radiation was very nearly the same as that of the nickel-plated copper surface. But at lower pressures an appreciable temperature difference developed. At 10^{-6} mm. Hg temperature equilibrium was reached and maintained at –30°C while the temperatures of the nickel-plated copper surface ranged from –198°C to 80°C. It is concluded that at comparatively high pressures heat is transferred by air conduction and convection between the dish and the nickel-copper plated surface, but at low pressures heat is transferred essentially by radiation. Calculations for the case of cooling to low temperatures confirm that heat (room temperature) supplied through the windows will balance the heat lost by the powder to the cold walls of the container when the temperature of the latter is about 50° below room temperature. The results of the experiments, shown in detail in a graph, indicate potential

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difficulties in the measurement of the luminescence glow curve where a linear heating rate is required and temperature equilibrium is not achieved.

Sommaire: Des expériences faites avec du phosphore sont rapportées et les résultats ont un intérêt dans le domaine des poudres isolantes pour vide.

Letter by R. H. Bube & R. E. Schrader *Rev. Sci. Instrum.* **25**, Sept. 1954 921-922

A New Type of Helium Liquefier 245/I

See Abstract No.: 169/II

A Linde Helium Liquefier-Cryostat Combination Suitable for Operation Down to 1.3°K 246/I

See Abstract No.: 170/II

The Organisation and Purpose of the Liquid Helium Pool 247/I

See Abstract No.: 69/IV

An Efficient Cryostat for Producing Temperature Between 4° and 80°K: The Production of Liquid Hydrogen Targets Using Liquid Helium 248/I

See Abstract No.: 193/III

16 — CHEMISTRY — 16

The Production of Radio-Isotopes 249/I

See Abstract No.: 187/III

Measurement of Temperature Distribution in a Low-Pressure Flat Flame 250/I

United States. The temperature distribution in a very lean, flat propane-air flame was measured by the thermocouple method, at a pressure of 0.0594 atmos. The temperature change occurred in a zone about 3 cm. thick. Wall-quenching was minimised by employing a burner tube diameter of 25 cm. The thermocouple was made from 12–micron wire, coated with ceramic to avoid catalytic effects. One hundred experimental traverse points were obtained, with a standard deviation of ± 0.016 cm. The burning velocity was also measured, and was found to vary with the inverse 0.30 power of pressure. This is in agreement with Egerton and Sen's result, obtained over a more limited range of pressure. The temperature pattern was analysed mathematically to obtain the pattern of heat release rate, taking into account the variation with temperature of thermal conductivity and specific heat. It was found that the heat release began at 300°C, attained a maximum value of 1.2 cal./cm.^{3}/sec. at 1,100°C, was nowhere negative, and was finite over a zone about 2 cm. thick. The maximum temperature of the flame was 1,332°C. The result obtained for the 'ignition point' was found to be extremely sensitive to the exponent giving the temperature dependence of the thermal conductivity. Some calculations relating to the chemical kinetics of the flame are described.

(*Science Abstracts*)

Sommaire: On a mesuré la distribution de température d'une flamme plate de propane et d'air, à la pression de 0.0594 atm.

Article by R. Friedman & E. Burke *J. Chem. Phys.* **22**, May 1954 824-830

Separation of Fluoranthene and Chrysene by Molecular Distillation 251/I

See Abstract No.: 182/III

Catalysis on Evaporated Metal Films III. The Efficiency of Different Metals for the Reaction Between Ethane and Deuterium 252/I

United Kingdom. The investigation reported here follows a previous investigation (Part II) published in 1953 (*Proc. Roy. Soc.*, **217A**, 376) which dealt with the reaction between deuterium and methane and was conducted with the object of determining the initial distribution of the various reaction products on a number of metal catalysts and the activation energies and frequency factors of the reactions together with the detailed kinetics. The catalytic apparatus employed in these experiments has been described in *Proc. Roy. Soc.*, **214A**, 1952, 413. A mass spectrometer of special design was used to analyse the gas mixtures. The method used for deposition of the metal films by vacuum evaporation has been described in Part I of the series of investigations. Orientation and structure of the films deposited was established by examination in the electron diffraction camera. Tungsten, molybdenum, tantalum, zirconium, chromium, vanadium, nickel, platinum, palladium, rhodium,

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ruthenium, manganese, silver, iron and cobalt films have been studied. All films were deposited at 0°C. The reaction vessel had a volume of 180 ml. The gas mixture admitted had an eight-fold excess of deuterium (4-fold for tungsten). The ethane component is shown in the table below. The initial distributions established

Catalyst	*Admittance Temperature*	*Est. No. of Ethane Mols.*	*Partial Pressure*
Tantalum, molybdenum, ruthenium, zirconium	−78°C	1.80×10^{19} mol.	2.32 mm. Hg
Tungsten	−78°C	2.25×10^{19} mol.	2.90 mm. Hg
Others	0°C	1.64×10^{19} mol.	2.58 mm. Hg

in the experiment are shown in a table partly reproduced below. In the case of molybdenum the main product

Distributions of Deutero-Ethanes for Various Catalysts

Catalyst	*Distribution*					
	d_1	d_2	d_3	d_4	d_5	d_6
Molybdenum	0.81	0.14	0.030	0.007	0	0
Chromium	0.47	0.18	0.066	0.060	0.072	0.15
Palladium	0.05	0.06	0.080	0.11	0.19	0.52

was C_2H_5D, in the case of palladium C_2D_6 and in the case of chromium both were major products. The activation energies obtained varied from 7.0 kcal./mole for molybdenum to a maximum of 21.4 kcal./mole for palladium. High frequency factors were found to coincide with high energies of activation. Manganese and silver films were completely inactive for exchange and cracking up to temperatures of 400°C. Iron was inactive for temperatures up to 370°C but showed, at about these temperatures, a pronounced tendency to cracking. Serious cracking of ethane occurred on nickel films above 160°C. The discussion of the experiments leads to the development of a theory for the likely mechanism accounting for the distribution. The process suggested is called 'repeated second point' adsorption proceeding in this particular case *via* adsorbed ethylene residues. The outcome of experiments on oriented and unoriented nickel films indicated that the nature of the crystal faces had an effect on the catalytic activities of the film.

Article by J. R. Anderson & C. Kemball *Proc. Roy. Soc.* **223A**, May 1954 361-377

Sommaire: L'éfficacité de differents métaux dans la réaction entre l'éthane et le deuterium a été étudiée comme partie d'une importante étude sur les performances catalytiques de films de métaux évaporés.

Determination of Moisture in Cereals: Review of Methods in Common Use

253/I

United Kingdom. The methods available for the determination of the moisture content in cereals are reviewed. No absolute measurements are possible, mainly due to the difficulty of defining the term moisture. A standard definition was attempted by Trolle (*J. Inst. Brewing*, 1949, 354) as follows:

> "By moisture shall be understood the loss in weight which the substance undergoes by evaporation during a reversible process of drying to practically constant weight, under such conditions that the substance does not undergo detectable alteration of any significance other than the reversible loss of moisture."

It is difficult to fulfil these conditions in routine determinations, and in practice, therefore, it is necessary to define moisture content in terms of the experimental procedure employed to determine it, *i.e.* the results of different methods do not necessarily agree. There are physical and chemical methods. Physical methods are: (*a*) *Oven drying*. This method is speedy, simple and gives reproducible results which are sufficiently precise for most commercial purposes. However, normal operational temperature is above 100°C and in some materials this temperature leads to chemical binding of the water and thus to low readings or, alternatively, decomposition of the material may be caused, yielding volatile decomposition products, producing high readings. (*b*) *Drying in vacuo*. In this method the temperature employed is governed by the requirement that the partial pressure of the water in the gas phase should be less than the vapour pressure of the water in the sample. The higher the vacuum, the lower the temperature required. Chemical drying agents employed in the process are listed in decreasing order of efficiency as follows: Phosphorus pentoxide, magnesium perchlorate, magnesium perchlorate trihydrate, fused caustic potash, calcium bromide, calcium oxide, and calcium chloride. The normal operational pressures are about 5 mm. Hg. In one instance quoted 0.1 mm. Hg was used. In that case maize grist was dried at 38°C, the whole test taking 340 hours. The ease of drying can be increased by passing a stream of nitrogen through the system. Several workers have reported that drying *in vacuo* at moderate temperatures (*e.g.* 40°C.) is more effective than drying at atmospheric pressure at 100°C. (*c*) *Distillation method*. The sample is heated with an excess of a liquid immiscible with water and the condensed vapours are collected in a graduated receiver; the volume of water is then measured directly. The method has the advantage that a constant temperature, determined by the boiling point of the liquid used, can be maintained indefinitely. The most important chemical method mentioned is the Karl-Fischer process. Certain recent modifications of this process are described. Methods of carrying out the titration and determining its end-point are discussed. See also Abstract 254/I.

Article by A. Bennett & J. R. Hudson *J. Inst. Brewing* **60**, Jan.-Feb. 1954 29-34

Sommaire: Les méthodes de détermination de la teneur en humidité communèment employées, sont revisées, afin d'être appliquées aux céréales.

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Study of Methods for Determination of Moisture in Malt

254/I

United Kingdom. Following up the survey reported in Abstract 253/I the results obtained with four commonly used methods of moisture determination were investigated, using malt as the test material. The samples were ground in a steel mortar prior to the tests. (1) *The Institute of Brewing Standard Water-Oven Method.* The malt was placed in flat tins and dried with the wall temperature of the oven at 98°C for various periods. It was found that constancy of weight was reached in approximately 3 hours. (2) *Vacuum drying.* The malt was placed in conical flasks suspended in a thermostatically controlled air-oven. A stream of dry nitrogen at a pressure of 0.5 mm. Hg was passed through the apparatus to increase the drying rate. Phosphorous pentoxide on pumice was used as the desiccant and the tests were conducted at various temperatures ranging from 40°C to 100°C. The samples were heated until there was no further loss of weight. It was found that the time required for a test decreased from 45 to 6 minutes as the temperature was raised from 40°C to 75°C. Between 75 and 79°C a sudden slowing down of the drying rate was observed indicative of a change in the nature of the mechanism involved in the liberation of moisture. (3) *Karl Fischer method.* Two methods were used to extract the water from the malt: (*a*) By stirring the sample with methanol at room temperature; and (*b*) by refluxing the malt with methanol for 10 minutes. (*a*) was found to take about 2 hours and (*b*) about 10 minutes. Determinations by method (*b*), however, always gave low values. (4) *Distillation method.* The determination was carried out in the Dean and Stark apparatus, using 100 ml. of liquid to extract 10g. of malt. Benzene and toluene were used as solvents; the latter was the more satisfactory. Difficulty was experienced in ensuring that all the water was collected in the receiving tube. The values obtained by the four methods are shown in a table, reproduced below.

Comparison of Moisture Content of Malts as Determined by Four Methods

Method	*Sample*						
	1	2	3	4	5	6	7
Institute Standard	2.91	2.94	2.87	3.03	2.87	2.80	2.90
Vacuum drying	3.46	3.64	3.68	3.39	3.56	3.56	3.59
Karl Fischer	3.75	3.69	3.70	3.60	3.61	3.90	3.70
Distillation method	3.5	3.6	3.4	3.7	3.9	3.8	3.9

The results obtained by methods (2), (3) and (4) were in satisfactory agreement, but method (1) gave consistently low values. The discrepancy may be due to enzymic hydrolysis of the starch or protein constituents resulting in chemical binding of initially free water. This may also account for the anomalous behaviour in the region 75–80°C when drying *in vacuo*. The method of differential thermal analysis was applied in order to demonstrate the occurrence of a chemical reaction during (1). In this method a change in the energy state of the reactants is evidenced by absorption or liberation of heat. With normal malt, an exothermic reaction, corresponding to enzyme action, was observed between 50°C and 80°C followed by an endothermic reaction at high temperature, due to the vaporisation of water. With black malt, which is manufactured in a manner precluding enzymic activity, only an endothermic reaction was observed.

Sommaire: Les performances des méthodes de détermination de la teneur en humidité, revisées dans le résumé 253/I sont étudiées, en employant le malt comme matériau de test.

Article by A. Bennett & J. R. Hudson *J. Inst. Brewing* 60, Jan.-Feb. 1954 35-42

Preservation of Bacteria

255/I

United Kingdom. The conventional way of preserving bacteria is to dry suspensions of the micro-organisms either from the frozen state or from the liquid state. A brief description is given of the performance and results of a new method which provides for the bacterial suspension to be introduced into material, already freeze-dried at this stage, and subjecting the resultant viscous product to vacuum drying. The freeze-dried material consists originally of a 6% peptone and 1% soluble starch solution sterilised in an autoclave. 1.5 micro-litre. of this mixture placed in 8 ml. glass ampoules was frozen to –20°C and subsequently dried for about 18 hours. The solids formed in the process had the shape of cylindrical plugs. The ampoules carrying the solids were connected to a manifold served by a rotary pump and fitted with a phosphorous pentoxide trap. Pumping was started and, by means of a needle valve, pressure in the manifold was adjusted at 10 mm. Hg. Inoculation was effected with the help of a capillary pipette. About 5 minutes after the last ampoule, out of about 12 to 18 connected to the manifold, had been inoculated and connected, the pressure was reduced from 10 mm. to 0.01 mm. Hg. Foam which had built up during pumping at the higher pressure was rapidly dehydrated after reduction to 0.01 mm. Hg. After a preliminary period of evacuation the ampoules were taken off, constricted and reconnected to the manifold ,and subjected to a final evacuation time of about 1 hour. Certain results obtained with the new method are given in the table partly reproduced below.

Organism	*Survival (%)*			
	1	2	4	8 *months*
Sh. flexneri	93	100	83	82
Salm. stanley	91	82	85	70

Sommaire: Description d'une nouvelle méthode pour préserver des bactéries, dans laquelle la suspension de bactéries est incorporée à un matériau lyophilisé auparavant et ensuite séché sous vide.

Letter by D. I. Annear *Nature* 174, 21.8.1954 359-360

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Abstract No. and References

Caution in the Use of Liquid Propane for Freezing Biological Specimens 256/I

See Abstract No.: 158/II

Viruses and the Electron Microscope 257/I

United Kingdom. Prior to the advent of the electron microscope it was not possible to see viruses. Their existence could only be proved by their action. The electron microscope facilitates the study of the morphology of viruses. Clear observation is greatly assisted by the use of shadow-casting techniques in specimen preparation. As a result the dimensions and shapes of many viruses are known today. The paper, concerned only with plant and insect viruses, mentions the tobacco mosaic virus which consists of rods about 290 mμ long and spherically shaped plant viruses such as the tomato bushy stunt and turnip yellow mosaic which are 27 and 20 mμ in diameter respectively, and finally the wound tumour virus of about 80 mμ diameter. Some details are given of the preparation of sections of virus-infected tissue, thin enough to allow the electron beam to penetrate them. Development and multiplication of viruses can now be observed. Details are given of various studies of the polyhedral diseases in Lepidoptera. There are two types of the disease, the 'nuclear disease' and the 'cytoplasmic disease'; the former develops in the cell nuclei of the skin, tracheae and fat body, the polyhedra being liberated into the cell finally reaching the blood, while in the case of the cytoplasmic type the virus originates in the cytoplasm and the polyhedra occurs in the tissue of the mid-gut. The article concludes with a short description of the virus causing the granular disease attacking tea plants. Each granule, itself of minute dimensions, consists of an outer capsule and an inner capsule, the latter holding the virus rod itself. Reference is made to the Leeuwenhoek lecture published in *Proc. Roy. Soc.*, **142B**, 1954, 196-207, which shows a number of electron micrographs demonstrating phenomena described in this article.

Sommaire: Revue des récents progrès dans la recherche de virus de plante et d'insecte, facilités par l' emploi du microscope electronique.

Article by K. M. Smith *Sci. J. Roy. Coll. Sci.* **24**, 1954 66-70

17 — METALLURGY — 17

Metallurgical Trends of Interest to Chemical Engineers 258/I

See Abstract No.: 215/I

Measurement of the Electrical Resistance of Metals and Alloys at High Temperatures 259/I

United States. Data on the solid-phase regions of the equilibrium diagrams of alloys at 1,500°C or above can only be determined by quenching the specimen and examining it in the microscope. The use of quenched samples has the disadvantage that phase changes often proceed too rapidly to permit sufficiently long retention of their high temperature form. Ambiguities in the measurements obtained by conventional methods could be eliminated by complementary measurements of other properties such as the electrical resistance of the material in that temperature region. A method of measuring the change of resistance with temperature of refractory metals and alloys up to their melting point is described in this article. The specimens used were rods or bars about 10 cm. long and 0.2–0.3 sq. cm. in cross section, having a hole about 1 mm. diameter and 4 mm. deep drilled into the bar near the centre to act as a black body for optical pyrometer measurements of temperature. A small furnace suitable for the pre-sintering of pressed powder bars consisting essentially of a tantalum or molybdenum tube is shown in an illustration. The specimens were directly heated by 60c/s current. The furnace employed for the measurements could be evacuated and consisted of a water-cooled base and water-jacketted cover made from brass or stainless steel pipe 7 inch. in diameter with a wall thickness of about $\frac{1}{8}$ inch. The current (up to 1,000 A) was fed in through hollow, water-cooled copper electrodes fitted to the furnace base. One end of the specimen was firmly clamped to its electrode while the other was maintained by a weight in sliding contact with the polished copper surface of the other electrode to allow for expansion. An observation port was provided to permit pyrometer observation, the port hole being covered on the inside by a second window which was moved out of the way while the measurements were taken, thus preventing the sight glass from being obscured by condensed vapours. Potential leads were spot-welded to the specimen bar. The potential difference between the leads was balanced against a fraction of that developed across a 1 ohm resistor connected to the secondary of a current transformer the primary of which carried the current used for heating the specimen. Balance was indicated by an a.c. galvanometer. A modified circuit for automatic recording of resistance *vs.* temperature is also described. The temperature was increased or decreased in small steps by a controller driving the variable auto-transformer which supplied the heating circuit. Details are given of a test run with tantalum as the specimen material. The temperature ranged from 1,100°K to the melting point. Prior to the test the sample was degassed for 30-40 minutes at a temperature of about 2,600°K. Pressure in the furnace was about 0.002 to 0.08 micron Hg. Values for the resistivity agreed to within 5% with those established by Langmuir and Malter.

Sommaire: On décrit une méthode de mesure des propriétés de résistance à l'electricité de métaux réfractaires et alliages a hautes températures, qui apporte des données complementaires qui ne pouvaient être obtenues sur des échantillons de ce matériau refroidis rapidement.

Article by P. Chiotti *Rev. Sci. Instrum.* **25**, Sept. 1954 876-883

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Exploratory Creep Test on Metals of High Melting Point

260/I

United Kingdom. In connection with gas turbine development it was desired to have some conception of the relative strengths, at a service temperature of 1,000°C, of possible construction materials, *i.e.* those with a melting point above 1,500°C. Accordingly a method of assessment, based on the measurement *in vacuo* of the compression strength at zero and at given intervals of time, was developed. The materials tested were titanium, zirconium, vanadium, niobium, tantalum, chromium, molybdenum, tungsten, platinum, palladium, rhodium, iridium, iron, cobalt, nickel, 80–20 nickel-chromium alloy, 'Nimonic 80', 'Stellite' with 9% tungsten and 29% chromium, 95% tungsten carbide bonded with 5% cobalt, tungsten-tantalum carbide bonded with 10% cobalt and titanium carbide bonded with 40% and with 50% nickel-chromium-cobalt alloy. Cylindrical test-pieces of $\frac{1}{8}$–inch diameter and $\frac{1}{4}$–inch length were employed. The ends were ground flat and parallel and the test-piece was compressed between two alumina push-rods. The specimens were tested in a small platinum resistance furnace evacuated to 0.02 mm. Hg at room temperature and subsequently heated by a battery to 1,000°C for 1–3 hours. The load was applied by a lever arrangement, the pressure being transmitted to the specimen through Sylphon bellows and recorded on a dial gauge. The temperature, which could be maintained constant within ± 5°C, was recorded by a thermocouple housed in a nickel-chromium alloy block surrounding the specimen set-up. Illustrations of the arrangement are given. The stress necessary to produce a strain of 1% in 24 hours was recorded. This percentage represents the approximate upper limit desirable in practice. Alternatively, a fixed load was applied and the temperature was stepped up by 50°C intervals, the creep being measured after ten minutes at each temperature. The composition of the metals tested is given in tables, parts of which are reproduced below

Material	*Composition in %*													
	Fe	*Si*	*Mg*	*Ca*	*Al*	*V*	*Cu*	*C*	*O*	*N*	*H*	*Nb*	*Ti*	*Ni*
Titanium: U.S. Bureau of Mines, Sheet	0.19	0.045	0.005	0.01	0.006	0.012	n.d.	0.064	0.18	0.027	0.002			
Vanadium: Ingot (Union Carbide)	0.082	0.075						0.075	0.085	0.017	—			
Tantalum: Fansteel	0.14	n.d.	n.d.	n.d.	n.d.		0.01	0.05				0.26	n.d.	
Tungsten: 5-mm. dia. rod	0.18		0.015	0.056	0.02		0.015	0.013					0.018	0.01

The strength of the pure metals at 1,000°C was found to vary from less than $\frac{1}{2}$ ton for titanium, zirconium, vanadium, platinum, palladium, iron, and nickel to as much as 6 ton/sq. inch. for tungsten and iridium. When plotted against the corresponding atomic numbers the values indicate a periodicity which has a parallel in that of the elastic constants at room temperature. The carbide materials were found to have a high creep strength at 1,000°C but show brittleness. The authors emphasise that the purity and history of the materials available were not in all cases accurately known. Thus the conclusions from the tests are somewhat tentative.

Sommaire: Une série de matériaux de construction pour haute température a été étudiée afin d'observer l'effet d'une force prédéterminée, appliquée sur l'échantillon pendant 24 heures à une température de 1,000°C.

Article by N. P. Allen & W. E. Carrington
J. Inst. Metals
82, July 1954
525-533

The Basic Factors Governing the Degassing of Metals in the Solid State

261/I

See Abstract No.: 216/I

Brewing Better Alloys in a Vacuum

262/I

United States. The scope of vacuum processing pure and alloyed metals is discussed from the commercial point of view. Vacuum metals production in the U.S.A. started approximately in 1944. By 1954 it had reached 30 tons per month and in 1955 it was expected to rise to 400 tons per month. Vacuum Metals Corporation and Utica Drop Forge and Tool Corporation are mentioned as the principal companies in the field. The former plans to increase its output from 20 tons per month in 1954 to 80 tons per month in early 1955. The latter with a production of 15 tons per month in 1954 hopes to reach 160 tons in 1955. Among the companies named as being in the process of starting up production of vacuum metals are the Universal-Cyclops Steel Corporation, The General Electric Company, Westinghouse and Crucible Steel. The size of the furnaces available for the vacuum production of metals has risen to a 1,000 lb. capacity per charge. The sudden rise in the demand for vacuum processed metals is due to the progressive realisation by the consumer industry of the practical and economical advantages offered by the use of vacuum processed metals in vacuum tubes, watch springs, gas turbines, transistors, tool steels, etc.

Sommaire: Discussion sur l'étendue commerciale des industries intéressées par le traitement sous vide des métaux.

Article by Anon.
Business Week
7.8.1954
110-112

I — General Science and Engineering — I
Contd.

Abstract No. and References

Method for Hi-Vac Alloying and Coated Product

263/I

United States. A process is described for the impregnation of base metals by applying a coat of elementary metal to the base metal in the solid state. In particular, the method aims at effective interstitial deposition of the impregnating metal on the base metal in order to secure a good bonding. The impregnating coat is deposited on the surface of the base metal by decomposition from metal carbonyls, facilitating coating of steel, cast iron and other metals by nickel, cobalt, chromium, iron, tungsten and molybdenum. The coating is carried out in a vacuum ranging from 18 to 23 inch. Hg in order to ensure that the metal surface, on which deposition takes place, is free from contamination normally present in the form of a monomolecular film and in order to remove all air retained in the interstices in the surface of the metal. Either liquid or gaseous carbonyls can be used in the process. By way of example the coating of the inner bearing surface of a cylindrical bearing is described. The component is placed in a vacuum chamber of circular shape and of a diameter appreciably larger than the bearing itself. A variety of outlets in the wall of the chamber connects to a vacuum pump *via* a freezing trap. Pumping is started and heat is radiated to the components by means of heat lamps fitted to the walls of the vacuum chamber, the component acquiring (and being kept at) temperatures ranging from 350–400°F. In order to prevent deposition of metal on the chamber walls the chamber is cooled and kept at a temperature of 250°F. The gaseous carbonyl, tungsten in the particular case, is kept at a temperature of 120°F in a gas accumulator under a pressure of 10–15 lb. per sq. inch. When suitable pressure and temperature conditions have been obtained in the chamber, the gaseous tungsten carbonyl is introduced into the chamber and directed towards the surface to be coated through jets in a distributing head which is positioned centrally in the vacuum chamber along the axis of the cylindrical component. On contact with the surface of the component the carbonyl is decomposed and tungsten is deposited at a rate of 0.01 inch per minute. In an alternative form the method provides for a lower vacuum and for an intermittent working of the vacuum pump, *i.e.* the vacuum is shut off during the actual coating period, and in this manner the final coating is built up stepwise. The degree of impregnation obtained in the process depends on the porous character of the surface of the base metal. The method is recommended as a means of providing rust protection, eliminating tarnishing and promoting surface hardening of metals, the latter being obtained by depositing tungsten onto the base metal.

Sommaire: Description d'une méthode de revêtement de la surface d'un métal de base par un procédé pyrolytique, afin de l'imprégner; cette méthode emploie le vide pour obtenir une bonne adhésion.

H. A. Toulmin, Jr. & Commonwealth Engng. Co. *U.S. Pat.* 2,685,124

Titanium Technology in Mid-1954

264/I

United States. An outline is given of the production, fabrication and mechanical properties of titanium and its alloys. Most titanium is produced from ilmenite by the Kroll process which, at one stage, requires the reduction of $TiCl_4$ with magnesium at high temperatures in an inert atmosphere. Magnesium chloride is removed from the resulting product by leaching and/or vacuum distillation, leaving commercially pure titanium. Since titanium is extremely reactive at high temperatures, it must be melted either in a vacuum or in an inert atmosphere. In the 'cold-mould furnace' the metal is arc-melted in a water-cooled copper mould. The whole unit is contained in a closed system which is evacuated and flushed with either argon or helium. Since the molten sponge freezes on coming into contact with the cold copper mould, the bulk of the melt, prior to solidification, is held in a shell of solid titanium minimising possible contamination from the mould material. Titanium has two crystal structures, α below 1,625°F and β between 1,625°F and the melting point, 3,135°F. Titanium alloys having good strength-to-weight ratios, creep and fatigue properties and satisfactory temperature resistance up to 1,000°F are produced in commercial quantities. Until the recent introduction of an α–type alloy all commercially available alloys showed both α and β structures. Development work is now in progress on pure β–alloys. Machining and grinding titanium is difficult because of its reactivity. Recommendations for grinding include the use of open-grain aluminium-oxide abrasive wheels, low wheel speeds, fast table speeds, and down feeds not exceeding 0.001 inch per pass. A grinding fluid should be used. For machining, slow speeds, heavy feeds and coolants or lubricants are required. The tool must be not allowed to ride on the work and chips should be cleared. Chatter should be prevented. Titanium components can be joined by heating *in vacuo*. This assists in eliminating the oxide skin. Welding, too, has to be carried out *in vacuo*. Welding to dissimilar metals produces brittle bonds, as does brazing. α–β alloys are quench-hardenable. β alloys are expected to have good bend ductilities. Deep drawing and stretch-forming can be carried out on titanium alloys but the metal must be worked as slowly as possible. At room temperature the creep strength of unalloyed titanium is about $\frac{3}{4}$ the yield strength, but at moderately high temperatures the creep strength is higher than the yield.

Sommaire: On donne un aperçu de la production, fabrication et propriétés mécaniques du Titane et de ses alliages.

Article by H. T. Clark J. P. Catlin & W. E. Gregg *Mech. Engng.* Sept. 1954 716-720

Some Observations Relating to the 'Wetting' of Titanium with Mercury

265/I

Australia. Mercury can be made to wet titanium after heating the titanium in a vacuum to about 700°C, so dissolving the oxide film on its surface, provided the mercury is brought into contact with the titanium under vacuum, and before the titanium has cooled below 400°C. On returning the mercury-wetted titanium to normal temperatures and pressures, the mercury starts to recede and after about 30 minutes the titanium surface will be free again of all mercury. Increased superficial brittleness of titanium treated as described was proved in bending tests, this brittleness apparently remaining even after the complete recession of the mercury.

Sommaire: On rapporte des phénomènes observés lors d'une tentative de mouillage du Titane avec du Mercure.

Letter by H. W. Worner *Bull. Inst. Metals* 2, Aug. 1954 147-148

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I — General Science and Engineering — I
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Abstract No. and References

Role of Beryllium in the Atomic Energy Programme

266/I

United States. In order to establish a controlled reaction in the fission of atomic fuels, a moderator is required to slow down the emergent neutrons to speeds at which they are capable of producing further reactions. This report describes the use of beryllium as a moderator and reflector in nuclear reactors. Three diagrams are given, showing the collisions of slow neutrons with fissionable nuclei, and the products of the reaction. Different types of reactor and the development stages in the design of a typical reactor are discussed. A table reproduced below shows relevant properties of the most commonly used moderators in thermal-type reactors. Beryllium would

Cross Sections for Thermal Neutrons

Moderator	*Density*	*For Scattering*		*For Absorption*
		Barns Per Atom*	*Barns per cc.*	*Barns per Atom*
Graphite, C	1.55 to 1.9	4.8	4.1×10^{23}	0.0045
Be Metal	1.845	6.9	8.5×10^{23}	0.009
BeO	2.2. to 2.9	11.1	6.7×10^{23}	0.0099
Heavy Water, D_2O	1.108	15.3	4.6×10^{23}	0.0009
Light Water, H_2O	1.000	44 to 164	15 to 55×10^{23}	0.64

* ▲ barn is 10^{-24} sq. cm.

be an efficient moderator if it were available in a form sufficiently pure to reduce neutron consumption by absorption. Beryllium is used as a neutron-reflecting medium in the Materials Testing Reactor of Arco, Idaho, a thermal reactor, operated with uranium as fuel, and light water as moderator and coolant. A sketch demonstrates the effect of a reflector, increasing the neutron flux density in the core of a reactor. A powder metallurgy technique was used to prepare pieces of beryllium in the desired shape. Commercially available beryllium pebbles were first vacuum-cast into ingots. The ingots were reduced to powder by turning on a lathe, and grinding the turnings in an attrition mill. Then a sintering process was used to obtain the final rough shapes of the desired dimensions ($4\frac{3}{8} \times 20 \times 64$ inch.) which were machined afterwards to close tolerances. The chemical specification for the beryllium was very rigid, and the impurity tolerances were given in terms of a few parts per million. It is concluded that, if beryllium was produced in more ductile form, its use in reactors would become more extensive. Ductile beryllium would also find other uses in industry since its strength-to-weight ratio is 30% greater than that of titanium. Extensive use would reduce the cost, at present still too high to attract the reactor builder. Prices to be aimed at are: Less than $ 25/lb. of raw beryllium of reasonable purity and $ 30/lb. for ductile beryllium ingots.

Article by R. E. Pahler *Metal Progr.* April 1954 86-91

Sommaire: On discute la part que le Beryllium peut jouer comme modérateur et réflecteur de neutron dans des réacteures nucléaires; certains aspects de sa production sont décrits et des références sont données sur son développement futur et son emploi étendu.

Preparation of High-Purity Lithium Metal by Vacuum Distillation

267/I

See Abstract No.: 174/III

Cathodic Vacuum Etching of Uranium

268/I

See Abstract No.: 168/III

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II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

High Vacuum Engineering 133/II
See Abstract No.: 206/I

High Vacuum Equipment 134/II
See Abstract No.: 205/I

The Measurement of the Pressure in the Determination of Pump Speed 135/II
See Abstract No.: 209/I

Simple Safety Device for Mercury Diffusion Pumps 136/II

United States. To prevent mercury vapours from reaching the vacuum system if an accident should occur and the cold trap cease operating, a thermostatically operated switch is placed in series with the heater of the diffusion pump, which will stop the pump if the trap temperature rises above a pre-set value. The authors have set the switch at –60°C and inserted the thermostat through the cork cover of the Dewar flask, so that the element was well immersed in the coolant. The device can be used for any coolant and does not affect the evaporation rate of the coolant.

Sommaire: Description d'un système de sécurité qui coupe le chauffage d'une pompe à diffusion au cas où le piège réfrigéré cesserait d'opérer.

Letter by W. S. Spindel & E. H. McLaren *Rev. Sci. Instrum.* **25**, Aug. 1954 830

Backstreaming of Oil Diffusion Pumps 137/II
See Abstract No.: 210/I

Pumping Speed Fluctuations in the Oil Diffusion Pump 138/II

United Kingdom. Pressure fluctuations on the high pressure side and on the backing side of diffusion pumps may have an important bearing on the effectiveness of leak detection methods. Details and results of measurements are given on such pressure variations occurring in a small diffusion pump of 3-inch. diameter fitted with two slightly varying jet constructions. The fluctuations determined in the experiments depend on variations of the heater input. The note concludes with a discussion of the results.

Sommaire: On a mesuré les fluctuations de pression existant du côté vide préliminaire et du côté vide moléculaire dans les pompes à diffusion.

Letter by C. S. Martin & J. H. Leck *Vacuum* **4**, Oct. 1954 486-489

Portable Laboratory Vacuum Pump 139/II

United Kingdom. The main disadvantages of providing a vacuum service in the form of water jet pumps in a radioactive laboratory are: (*a*) That the water must be treated for contamination, and (*b*) the system must be situated in the vicinity of a water supply. Two alternative designs of a self-contained unit, mounted on a trolley, were developed to overcome these disadvantages. The first, consisting of a rotary mechanical pump and auxiliary equipment facilitating the circulation of the water in a closed circuit, was rejected on account of the cost. The second system makes use of a water jet pump mounted in a light alloy casing, the water being circulated through the jet from a reservoir by means of a centrifugal pump. If produced in reasonable numbers the cost of the latter unit is about £50. Trials with prototype units indicate a maximum service life, without maintenance, of about 350 hours when operated continuously; the lowest pressures obtained were 74 cm. Hg at room temperature and 68 cm. Hg at water temperatures of 70°C. Intermittent operation lengthened the service life. In all cases, failure was due to worn brush gear. The vacuum obtained at 70°C water temperature could be improved by charging the reservoir with equal quantities of water and ethylene glycol. Frothing occurred when the system was opened to atmosphere but could be eliminated by the addition of a silicon anti-foam agent.

Sommaire: Ce rapport décrit le dévelopement d'une pompe à vide portable, à injecteur, pour être employée dans les laboratoires atomiques.

Report by A. Bury *A.W.R.E. Rep. No.* O-4/54 January 1954

Ultimate Vacuum in a Vacuum-Enclosed Ionisation Gauge 140/II
See Abstract No.: 143/II

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

On a New Device for Evacuation in High Vacuum — 141/II

Japan. A glass bulb of 15 cm. diameter, furnished with a barium source and a hot cathode Philips gauge type discharge tube, was found to serve as an excellent high vacuum pump for oxygen, hydrogen, carbon dioxide and water vapour. Pumping speed of about 80 l/sec. at 3×10^{-7} mm. Hg was recorded for oxygen, 8 l/sec. at 2.5×10^{-6} mm. Hg for hydrogen, 50 l/sec. at 1.2×10^{-7} mm. Hg for carbon dioxide and 26 l/sec. at 2.5×10^{-7} mm. Hg for water vapour.

(*Science Abstracts*)

Article by O. Tsukakoshi *J. Sci. Res. Inst. Japan* 47, Dec. 1953 133-148

Sommaire: Description d'un nouveau principe de pompage, basé sur l'action d'une source de barium en liaison avec une jauge Philips de type tube à décharge.

Removing Carbon Bisulphide from Viscose Fibre with a Liquid Seal Vacuum Pump — 142/II

See Abstract No.: 196/III

21 — GAUGES — 21

Ultimate Vacuum in a Vacuum-Enclosed Ionisation Gauge — 143/II

United States. Details are given of experimental work carried out to check the theory that the lowest pressure attainable in a Bayard-Alpert type ionisation gauge operating as a pump (about 5×10^{-11} mm. Hg equivalent nitrogen pressure) is limited by the diffusion of atmospheric helium through the glass walls of the gauge. The gauge investigated was completely enclosed by an outer glass envelope connected to a separate vacuum system. The lowest pressure obtained with this arrangement was estimated to be substantially less than 10^{-11} mm. Hg, after allowing for the spurious collector current due to the emission of soft X-rays from the grid. It is shown that the X-ray current may be appreciably reduced by lowering the grid potential from 150 to 70 V.

Letter by L. J. Varnerin Jr. & D. White *J. Appl. Phys.* 25, Sept. 1954 1207-1208

Sommaire: On a étudié l'effet de diffusion de l'hélium atmospherique au travers des parois de verre d'un système porté à un vide trés poussé.

Compound Mercury Manometer — 144/II

United States. In the course of experiments on filtration, pressures up to 75 p.s.i. were measured by placing a number of mercury manometers in series. This arrangement permits greater accuracy of measurement than that obtained with Bourdon gauges. The pressure on the last manometer is the original pressure less the sum of the pressure drops in the preceding manometers. If air is left above the mercury the pressure drop differs from tube to tube because of the changes in absolute pressure but if a liquid is placed above the mercury the pressure drop is the same in each instrument as long as the diameter of the gauge tubes is the same. Only one of these identical values need be determined and the last manometer can be calibrated to give a direct reading. By a suitable arrangement any number of manometers constituting the 'compounded' instrument can be brought into use to suit measurement in the desired pressure range. However, the larger the number of manometers used in series, the lower is the accuracy of the readings. Accuracy can be improved by employing an inclined or spiral manometer for the last unit. The compound unit can serve as a barometer or vacuum gauge, if the low pressure side is sealed off.

Note by F. M. Tiller *Analyt. Chem.* 26, July 1954 1252-1253

Sommaire: Description d'une méthode de mesure des pressions de filtration, jusqu'a 75 livres, au moyen de manomètres à mercure qui peuvent être rapidement modifiés pour mesurer des basses pressions.

A Universal Apparatus for High-Precision Pressure Regulation at Reduced Pressures — 145/II

Poland. The universal apparatus is a modification of Todd's apparatus (cf. Chem. Abstracts 43, 1226*d*). It consists of 2 parts of which one, containing monostatic fluid (*e.g.*, Hg, oil, H_2SO_4, esters), prevents the increase and another, containing an electrolyte, the decrease in the pressure. It works in connection with an electronic device for pressure regulation in the range of 0–100 mm. Hg. The stability of the pressure in the assembly depends on the use of monostatic liquid and on the air-tightness of the apparatus. At a pressure 10–20 mm. Hg and gas quantity 50–200 c.c./min. the exactness of the measurements is within 0.01–0.1 mm. Hg. The drawing and the picture of the apparatus are given.

(*Chemical Abstracts*)

Article by M. Bukala & J. Majewski *Przemysl Chem.* 8, 1952 510-512

Sommaire: Discussion d'une modification apportée à l'appareil de Todd servant comme régulateur de pression, de haute précision, dans la région de 10 à 20 mm. Hg.

22 — MEASURING PLANT (VACUUM) — 22

A High-Temperature Microwave Spectrometer — 146/II

See Abstract No.: 242/I

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II — Vacuum Apparatus and Auxiliaries — II

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Abstract No. and References

A Mass Spectrograph for the Analysis of Solids

147/II

United States. A spark-source mass spectrograph of the Mattauch-type is described, designed for the analysis of minute concentrations of impurities, a problem made acute by recent developments in semi-conductors. The spark is obtained between two short wire specimens (0.030–0.040 inch in diameter) held in pin chucks, and energised from a pulsed 800 kc/s source at 50–75 kV. This scheme localises the spark to the specimen. The ions produced are subjected to both direction and velocity focusing. After passing through a tantalum accelerating plate and two tantalum slits, they enter the electrostatic analyser in which they are deflected through about 32° at a radius of 15 cm. The accelerating voltage is about 19 kV, obtained from an r.f. source and the deflection voltage is 1,440 V, obtained from batteries. The beam then passes through a third slit into the magnetic analyser where it follows a curved path in the $\frac{1}{8}$-inch gap between the two gold-plated pole pieces After deflection through about 90°, the dispersed beam falls on a photographic plate. Alternatively, the ions may be allowed to pass out of the magnet gap and enter an electrical collector mounted in a side tube. The source chamber and electrostatic analyser chamber are made of O.F.H.C. copper, the joints being oven-brazed with copper-silver eutectic, except for the demountable flanged joints which have Neoprene gaskets. Pumping tubes, electrical connections and spark observation windows are made from O.F.H.C. copper tubes oven-brazed into the chamber wall, tubular Kovar glass seals then being r.f. brazed to the copper tubes. The magnetic analyser is made of gold-plated iron with O.F.H.C. copper spacers and has a Kovar glass pumping tube. The magnet (core section 5×8 inch., two coils of 27,500 turns each) gives a field of 18,000 oersted in the $\frac{1}{8}$-inch gap. Separate 2-stage mercury diffusion pumps are provided for source chamber and analyser, each pump having two liquid nitrogen traps. A dry-ice trap is provided between the common backing pump and the diffusion pumps. Each high-vacuum line has a glass 'valve', which is shown in an illustration, consisting of a hinged glass plate which can be tilted by external magnets so as to fall on to a silicone rubber seating on the $1\frac{1}{2}$-inch. pumping orifice. These arrangements enable source and plate to be changed without cooling the diffusion pumps. Within 2 hours, after this operation, the pressure will be down to 10^{-6} mm. Hg and falls further to $2\text{–}4 \times 10^{-7}$ mm. Hg in a few hours. The resolving power of the instrument is about 1 in 3,000 and it is estimated that concentrations of about 0.1 p.p.m. (atom fraction) can be detected, or about 0.1 monolayer in the case of surface contamination. Calibrations have shown semi-quantitative results in the concentration range 10^{-7}–10^{-4}.

Sommaire: On donne des détails sur un spectromètre de masse à double focalisation pour l'analyse de petites concentrations d'impuretés dans des solides.

Article by N. B. Hannay *Rev. Sci. Instrum.* **25**, July 1954 644-648

A High-Resolution Evaporated-Carbon Replica Technique for the Electron Microscope

148/II

United Kingdom. There are two basic types of electron microscope replicas: (*a*) Consisting of a film which has a flat surface remote from the specimen surface, and (*b*) consisting of a film of uniform thickness following the topography of the specimen surface faithfully. Typical of (*a*) is the Formvar replica; typical of (*b*) is the silica replica. The Formvar replica is reputed to have disadvantages from the optical point of view whereas preparation by the silica method is known to take a long time. A new method is described employing carbon as a final replica and using Formvar only during intermediate stages. In this method a 1–2% solution of Formvar is first poured on to the surface of the specimen and the thin film thus produced is backed by a layer of a soluble resin called 'Bedacryl'. When the Bedacryl has hardened, the compound film is stripped off with the aid of Sellotape. A film of carbon is then deposited on the Formvar side of the compound film, which carries the impression of the surface to be examined. This deposition is effected by evaporating carbon *in vacuo* from two pointed, 5 mm. rods of 99% hard graphite, a current of 20–30 A being passed through the rods, the points of which are kept in light contact. The progress of the deposition of the carbon is followed by observing the discoloration of a piece of originally white glazed porcelain placed alongside the replica being coated. A film thickness of about 50 Å can be detected by this means. Three methods of dissolving away the resin and mounting the carbon film are described; two of these methods permit shadow-casting of the carbon film. Micrographs obtained from the replicas are reproduced, the good resolutions and clarity being attributed to the ability of the Formvar to accurately follow the surface topography and yet strip off without distorting, while the carbon film is stable and has good electron-optical properties. A carbon film replica can be made in about 15 minutes. An important advantage is that it is readily visible during processing without special lighting.

Sommaire: Description d'une technique de replica en deux opérations, employant un film Formvar intermédiaire et un film de carbone évaporé.

Article by D. E. Bradley *J. Inst. Metals* **83**, Sept. 1954 35-38

Viruses and the Electron Microscope

149/II

See Abstract No.: 257/I

Decompositions of Inorganic Specimens During Observation in the Electron Microscope

150/II

United States. It has been repeatedly reported in the literature that specimens of substances examined in the electron microscope decompose or suffer other alterations under the impact of the electron beam and the influence of the rarified atmosphere in the chamber. Reference is made to reports on such phenomena observed with crystals of sodium chloride, sodium chlorate, potassium bromide, etc. The present investigation, aimed at obtaining further information of such phenomena, is concerned with (1) sodium chloride and potassium chloride, (2) ammonium chloride and nitron nitrate, (3) silver chloride, (4) silver nitrate and sulphate, cupric oxide, gold chloride, lead carbonate, oxide and chloride and (5) hydrous ferric oxide and aluminium oxide.

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Selected area diffraction techniques were employed for the tests which were carried out in an R.C.A. type E.M.U. electron microscope fitted with an electron diffraction attachment. Parlodion-covered grids were used to carry the specimens which were either applied in dry form (powder) or by evaporation in air or vacuum. Large sodium chloride crystals, opaque at the outset, developed transparent envelopes, outlining the original size of the crystal and containing a multitude of smaller crystals of the same substance, during the examination. Potassium chloride crystals showed a similar tendency of breaking up but only as long as the electron beam was *passing* through the specimens, *i.e.* when the instrument was used as an electron microscope. This suggests that heat may be an important factor. Ammonium chloride evaporated, and nitron nitrate melted. Silver chloride was reduced to free silver. Also appreciable sputtering of the silver was noticed. Silver nitrate, cupric oxide and lead carbonate were all reduced to their free metallic component. But silver sulphate, gold chloride, lead oxide and lead chloride showed only morphological changes. The mechanism involved is not clear. Direct electron beam reduction as well as photo-chemical reduction seems to be involved. Finally, hydrous ferric oxide and aluminium oxide were transformed into unidentified crystalline structures during exposure to the beam.

Sommaire: On donne des détails sur les changements observés dans des spécimens de différentes substances non-organiques, soumises au faisceau d'un microscope électronique.

Article by R. B. Fischer *J. Appl. Phys.* **25**, July 1954 894-896

Cathodic Vacuum Etching of Uranium 151/II

See Abstract No.: 168/III

An Electron-Diffraction Examination of Thin Films of Lithium Fluoride and Copper Prepared by Vacuum Evaporation 152/II

See Abstract No.: 159/III

A Torsional Pendulum of Low Thermal Inertia 153/II

United States. A torsional pendulum is described, constructed to study damping in certain gold-nickel alloys, in particular the relaxation effect which occurs when the alloys are quenched from high temperatures. Because this effect disappears rapidly a low thermal inertia instrument is required. In the new design of a pendulum described in this note this requirement is satisfied by abandoning the conventional principle of heating the specimen externally and heating the specimen, instead, by passing an electric current through it. Details of the new design are given in an illustration. The specimen in the form of a 0.030 inch diameter wire 10 cm. long, forms the suspension of an inverted torsional pendulum, counter-balanced to minimise longitudinal stress. Initially, difficulty was experienced in keeping the temperature along the wire uniform as the clamps at the end of the wire acted as heat sinks, but in later designs uniformity of heat within 5°C at 350°C over a length of 7 cm. of the wire could be maintained successfully. The whole pendulum is encased in a metal bell jar with a suitably placed viewing window, the bell jar being evacuated prior to measurements in order to counteract oxidation at high temperatures.

Sommaire: Description d'un pendule à torsion, opérant dans une chambre à vide, developpé pour étudier certains phénomènes métallurgiques.

Letter by C. Ang & C. Wert *J. Appl. Phys.* **25**, Aug. 1954 1061

26 — MATERIALS — 26

Rubber in Vacuum Systems 154/II

United Kingdom. It has been noticed that copper employed in a vacuum system suffers discoloration if rubber of certain types is present. The phenomenon has not been observed in the presence of natural rubber or silicone rubber nor has it been observed on evaporated copper films. Discoloration is only apparent if the copper is present in the form of a solid.

Sommaire: On a observé une décoloration du cuivre employé dans un système à vide, en présence de quelques types de caoutchouc, bien que le caoutchouc naturel ou au silicone semble être inoffensif.

Letter by J. G. S. Biram & T. A. J. Jaques *Vacuum* **4**, July 1954 341-342

Gasometric Method for Determination of Hydrogen in Carbon 155/II

United States. A low pressure method is described of measuring hydrogen contents of 0.0004 to 3.5% in carbon. Samples of the carbon to be examined are burned in an oxygen stream of about 4 cm. Hg pressure. The water formed during the combustion is condensed in a dry ice freezing trap. After the remaining gases have been pumped away the water is released from the trap and transferred in the form of water vapour to a calibrated, closed-end manometer, where its vapour pressure is measured. The corresponding hydrogen content is determined by calculation. The experimental equipment is made of borosilicate glass throughout, except for the oxygen purification section. No stopcocks are used, except for the admission of oxygen to the purification train, and in the pumping line. Instead, mercury cut-offs are employed, operated by a separate vacuum pump.

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Essentially, the plant consists of a purification train embodying a catalyst and a nitrogen trap, a furnace section incorporating provision for storing a limited number of samples under vacuum, and the actual furnace, the necessary heating being obtained by a high frequency unit consisting of a vacuum tube oscillator with output less than 2 kW and operating at about 500 kc, a section containing two double freezing traps (dry ice) in series, the analytical section comprising a McLeod pressure gauge and two calibrated manometers, and, finally, beyond the last mercury cut-off, the pumping line and auxiliary apparatus connecting to a diffusion pump unit. The samples to be tested consisted of carbon granules, a weighed amount of which was placed in small bags 10 mm. high and 7 mm. diameter made from 150-mesh platinum gauze. Up to 12 such bags were sealed into the vacuum system prior to the tests. In operation, the whole of the apparatus is evacuated as a first step. After an initial pumping period the oxygen purification train is shut off and the catalyst heat-treated in preparation for the test. During this period, and sometimes for up to about 7 hours, pumping of the remainder of the system continues down to a pressure of 2.3×10^{-5} mm. Hg in order to obtain thorough outgassing of the system and the carbon samples. Then connection is re-established with the oxygen purification train and oxygen is admitted to the system at a pressure of 4 cm. Hg. At the same time a carbon sample is passed into the platinum crucible and heated at 1,000°C for ten minutes. Then the furnace is switched off and the oxygen supply cut off, while pumping continues. In order to avoid removal of water vapour by the pumps, the mercury in the last cut-off is raised to a point where only a capillary passage is available for the exhaustion of the system. When the McLeod gauge indicates satisfactory removal of all residual gases, pumping is stopped and the cut-off in the pumping line closed completely. Then the water condensed in the freezing traps is removed in the form of water vapour to the calibrated manometer by transferring the dry ice bath from the traps to the manometer side arm. After raising the mercury to the given calibration mark on the manometer the dry ice bath is removed. After the water vapour has reached room temperature (25°C) the pressure is measured. In the present experimental set-up two manometers are used, each with three calibration marks indicating volumes of 130, 140, 150 and 20, 30 and 40 ml. After having established volume (V_1) and pressure (P_1) for the water vapour obtained as a blank, the hydrogen content of the sample can be determined from

$$\frac{(P \text{ mm.} \times V \text{ ml.})^{25°C} - (P_1 \text{ mm.} \times V_1 \text{ ml.})^{25°C} \times 0.1084 \times 100}{\text{weight of sample } (\gamma)} = \% \text{ hydrogen}$$

Article by W. G. Guldner & A. L. Beach *Analyt. Chem.* **26**, July 1954 1199-1202

Sommaire: Description d'une méthode pour mesurer à basse pression des petites quantités d'hydrogène, de 0.0004 jusqu'à 3.5% dans le carbone.

Adsorption of Helium on Glass at Temperatures Below the λ –Temperature 156/II

See Abstract No. : **67/IV**

Diffusion Coefficient, Solubility, and Permeability for Helium in Glass 157/II

United States. Reference is made to a number of investigations published in the past on permeation of different types of glass by various gases. Williams and Ferguson (*J. Amer. Chem. Soc.*, **44**, 1922, 2160), investigating the permeability of Pyrex for helium, were the first to draw attention to the relationship between permeability, solubility and diffusion coefficients, and published the only solubility value so far available, = 0.008 at 515°C. The three quantities are related in the form k = DS, where k is the permeability, S the solubility and D the diffusion coefficient. Dividing previously published permeability figures by the solubility value just mentioned gives inconsistent values for the diffusion coefficient, and this caused the authors to develop a method whereby the three qualities can be measured simultaneously for a given gas in a single sample of solid material, as described in this article. The theoretical basis of the new approach is discussed in great detail. Expressions are obtained from the fundamental diffusion law (Fick's law) which enable calculation of the diffusion coefficient from pressure measurements taken either in the early part of the transient period (method of early approximation) or in the period of steady state gas flow (method of later approximation). The method is greatly facilitated by the availability of modern ultra-high vacuum techniques which ensure the absence of leaks and contamination, and permit operation with very small quantities of gas. The authors used a Pyrex system in their experiments with helium as the test gas. Details of the experimental set-up are given. The part of the system into which the helium diffused was finally evacuated by ion gauge pumping. The experimental data required for the determination of the quantities were recorded in terms of pressure indication against time. Experimental values of diffusion coefficient obtained for various temperatures and pressures are given in tabular form. The relation DS=k has been confirmed. The diffusion coefficients and permeability values obtained in the experiments show temperature dependence but the solubility shows little variation between 25°C and 300°C and agrees well with the value established by Williams and Ferguson.

Article by W. A. Rogers, R. S. Buritz & D. Alpert *J. Appl. Phys.* **25**, July 1954 868-875

Sommaire: On donne des détails sur une méthode de détermination simultanée des coefficients de diffusion de solubilité et de perméabilité d'un gaz donné, ceci dans un échantillon d'un matériau solide.

Caution in the Use of Liquid Propane for Freezing Biological Specimens 158/II

United States. Liquid propane has recently been brought into use for rapid freezing in connection with the freezing and drying of biological specimens. The use of this material carries with it the possibility of an explosion when cooling is effected by means of liquid nitrogen in a vapour trap open to the atmosphere. Propane melts at –189.9°C, oxygen liquifies at –183°C. Thus when operating in this range of temperatures

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it is possible to get a mixture of liquid oxygen and liquid propane. The following precautions are advised: (1) Provision of a rapid flow of vapour into the condenser to exclude air and (2) to keep the temperature of the materials above the boiling point of oxygen.

Sommaire: Description des risques existant dans l'emploi de propane liquide pour descendre à des températures voisines du point d'ébullition de l'oxygène.

Letter by J. L. Stephenson *Nature* 174, 31/7/1954 235

A New Method of Sealing-off Quartz Vessels in Vacuo

159/II

Israel. Normally quartz glass tubes are sealed off by an oxygen-hydrogen flame. This has the disadvantage that hydrogen is liberated and diffuses into the glass. A new method of sealing off quartz vessels *in vacuo* is described which overcomes this disadvantage by employing an arc. A suitable tool to facilitate the method is described.

Sommaire: Description d'une nouvelle méthode employant un arc electrique, pour fermer sous vide des enceintes de quartz.

Letter by B. Schneidmesser *Vacuum* 4, October 1954

A Simple Method of Making Vacuum-Tight Coolable Window Seals for Low Temperature Optical Transmission Cells

160/II

See Abstract No.: 167/II

Linings for Induction Furnaces

161/II

See Abstract No.: 179/III

Behaviour of Refractory Metals, Single or in Contact with Each Other, At High Temperatures in a Vacuum

162/II

United States. The manufacture of vacuum high temperature apparatus very largely depends on the availability of refractories showing a satisfactory stability under the operating conditions envisaged for the apparatus. These refractories may be used either as normal construction material or to serve as a base and support for the components processed in the apparatus. The investigation reported here is concerned with their properties, (*a*) when used as a construction material and (*b*) when in contact with another refractory under high temperature in vacuum. The following materials have been investigated: Beryllia, magnesia, zirconia, thoria, molybdenum, tungsten and graphite. The temperature ranged from 1,500 to 2,300°C; the specimens had the form of small cylinders, produced by dry pressing. The experiments were carried out in an induction furnace described in *J. Amer. Ceram. Soc.*, 1949, 316. Changes of density, volume and weight of the specimens were determined in one series of the experiments and in a second series reactions resulting from intimate contact between two refractories were examined. The results of the first series of experiments are given in graphs, and the results of the second series are given in a table partly reproduced below. The

Combination	*Reaction starts at °C*	*Nature of the Reaction*	*Remarks*
Carbon-Tungsten ...	1,500	Formation of carbide	
Carbon-Zirconia... ...	1,600	Reduction of the zirconia	Carbide formation at high temperatures
Beryllia-Thoria	2,100	Melting	Complete liquefaction at 2,200°C
Magnesia-Molybdenum...	1,600	Silver coloured deposition on magnesia	No erosion of magnesia, considerable erosion of molybdenum at 2,100°C
Zirconia-Thoria	2 200	Little adhesion	No liquid phase at 2,300°C
Molybdenum-Tungsten	2,000	Little adhesion as a result of sintering	Better adhesion at 2,300°C

outcome of the experiments permits the following conclusions: (1) In a vacuum of the order of 10^{-4} mm. Hg and at high temperatures the factors governing the uses of refractories are not the melting point of the material but (*a*) its stability when in contact with other materials and (*b*) its evaporation characteristics at that temperature level *e.g.* Magnesia, in a vacuum, can only be exposed to temperatures at about 1,700°C for short periods. Beryllia is unsuitable for operation at any temperature above 2,100°C. But carbon, zirconia, thoria, molybdenum and tungsten can safely be exposed to temperatures up to 2,300°C for protracted periods. (2) Certain oxides, such as magnesia and beryllia show incipient evaporation at temperatures as low as 1,600 and 2,000°C respectively. The experiments indicated that the use of an inert atmosphere effectively counteracts evaporation. (3) Beryllia showed the greatest stability of all oxides tested in contact with carbon, zirconia had a better stability than others in contact with molybdenum, and thoria showed the greatest stability of all oxides in contact with tungsten. (4) The most stable combinations of oxides were zirconia—thoria and magnesia—thoria.

Sommaire: On a étudié les changements et propriétés physiques ansi que les réactions chimiques qui prennent place lorsque des matériaux réfractaires sont employés dans des appareils à vide, ceci dans différentes conditions.

Article by P. D. Johnson *Ber. Deutsche Ker. Ges.* 31, No. 3, 1954 81-84

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Some Observations Relating to the 'Wetting' of Titanium with Mercury 163/II

See Abstract No. : 265/I

28 — APPARATUS COMPONENTS — 28

A Pressure-Vacuum Diaphragm Valve 164/II

United Kingdom. Reference is made to a valve originally designed by Halban and Wilson (*J. Sci. Instrum.*, **24**, 1949, 414), employed in the thermal diffusion plant. The valve is required to withstand high pressures, have a low flow resistance and, in addition, to be vacuum tight. The original design suffered from three drawbacks; diaphragm wear, inability to stand pressures above about 500 p.s.i., and moulding of the valve diaphragm which prevented the valve from opening when used as a vacuum valve. Details are given of the measures taken to improve the performance of the valve. The diaphragm material used in the old valve, *i.e.* natural rubber Dunlop EX3007 without canvas backing, was replaced with Hycar E75 (Gaco) material of 1/16 inch nominal thickness. Other diaphragm materials tested were Neoprene, natural rubber and Perbunam. Details of the tests are given. Neoprene may be preferable in cases where it is required to admit gas slowly into a sample system. The diameter of the diaphragm is 7/8 inch. The diaphragm acts as both seat and seal. The valve is shut by forcing the diaphragm by means of a plunger onto a circular ridge, and it is opened by returning the diaphragm by means of a spring. The valve is 2 inch. long and hexagonal in cross section, the flats being 1.2 inch. across. When closed, the valve has an internal volume of about 1 c.c. The new design operates safely at pressures up to 2,000 p.s.i. but the operation of the valve between 1,000 and 2,000 p.s.i. is difficult. The leak rate across the valve seat with 1,500 p.s.i. on one side and vacuum on the other is less than 10^{-4} l. μ/sec. The valve is suitable for pressure systems requiring a low flow resistance and which need to be evacuated occasionally through the valve. Full drawings with details of general assembly are given and a complete inspection schedule is included.

Sommaire: On donne des détails sur la réalisation et les performances d'une vanne à vide, équipée d'un diaphragme entièrement supporté et enfermé.

Report by R. W. Bowring & R. H. Davies *A.E.R.E. Rep. No. GP/R.* 1481 August 1954

Improvements Relating to Diaphragm Valves for Controlling Small Gas Flows 165/II

United Kindgom. An on-off type valve is described which is used for controlling small predetermined gas flows. It comprises a body section and a cover plate screwed together, with a diaphragm of flexible metal positioned between the two components, and a rubber or soft metal sealing ring provided in the body section to ensure gas tightness of the joint. The inlet and outlet ports in the body section communicate *via* a small cavity beneath the diaphragm. The inlet terminates in a short tube, mounted on the axis of the diaphragm, which contains a porous plug to restrict the gas flow to the desired maximum. The clearance between the diaphragm and the end of the tube is of the order of 0.005 inch, and to effect closure the diaphragm is forced against the tube by a screw located in the cover plate, acting *via* a pressure pad. The tube carrying the porous plug can be easily exchanged and it is visualised that a number of such tubes (duct units) should be available, calibrated for different maximum flows. For automatic operation the screw may be replaced by a solenoid-operated plunger.

Sommaire: On donne des détails sur une vanne pour controler de minces écoulements de gaz dans des systèmes à vide.

K. Heathcote, J. Blears & Metropolitan-Vickers Electrical Co. Ltd. *Brit. Pat.* 712, 856

A Simple Magnetically-Controlled Mercury Cut-Off 166/II

United Kingdom. A simple U-shaped mercury cut-off, especially designed for use in gas-flow systems, is described. One arm of the U consists of a large diameter tube, housing a steel slug in a glass envelope floating on mercury. Immersion of the slug in the mercury is effected magnetically. The other arm of the U, of smaller diameter, leads to the bottom limb of a vertical Y junction in the vacuum system. On immersion of the slug mercury rises into the upper two limbs of the Y and isolates the two parts of the vacuum system.

Sommaire: Description d'un interrupteur à mercure spécialement développé pour être employé dans des systèmes à circulation de gaz.

Note by V. J. Hammond *J. Sci. Instrum.* 31, July 1954 258-259

A Simple Method of Making Vacuum-Tight Coolable Window Seals for Low Temperature Optical Transmission Cells 167/II

United Kingdom. In experimental work involving the irradiation of specimens at low temperatures, the chamber housing the specimen requires windows which have to be sealed to the chamber in a manner ensuring vacuum tightness at the desired low temperatures. Normally the materials of the windows are different from those preferably employed in the construction of the vacuum chamber, *i.e.* glass and copper. Thus special sealing techniques have to be applied. A sealing method which has proved satisfactory is described in this article. The window in the form of a thin disc is placed on an annealed copper diaphragm 0.003 inch thick with a hole in the centre of a diameter $\frac{1}{8}$-inch less than the window. The angle between the disc and the copper

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diaphragm is filled with Araldite powder (type 1) and the assembly heat-treated for one hour at 190°C. Finally the assembly is affixed to the copper tube, forming the specimen chamber, by means of Wood's metal. Araldite has proved satisfactory at low temperatures and has a low vapour pressure. It also has sufficient flexibility to take up some differential expansion, but it is unsuitable for prolonged experiments above 150°C owing to decomposition. The following window materials have been tested in connection with the window design described.

No.	*Material*	*Thickness (inch.)*	*Diameter (inch.)*
1	Glass	0.05	1
2	Fused silica	0.09	1
3	Synthetic sapphire (Al_2O_3) ...	0.02	0.75
4	Synthetic fluorite (CaF_2) ...	0.07	1
5	Silver chloride	0.06	1
6	Thallium bromo-iodide (KRS5)	0.1	1

Window assemblies with any of the materials 1–5 can be cooled rapidly to 77°K, assemblies with 1, 3 and 5 can be cooled down to 20°K. Assemblies with material 6 will remain vacuum-tight at 77°K if cooled down slowly and with great care but cooling down to temperatures as low as 20°K results in the window becoming almost opaque due to the formation of polycrystals.

Sommaire: Description d'une nouvelle méthode de fermeture, appliquée aux fenêtres de cellules de transmission optique, employées dans des recherches sur les basses températures.

Article by V. Roberts *J. Sci. Instrum.* **31**, July 1954 251-252

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29 — MISCELLANEOUS APPARATUS — 29

The Organisation and Purpose of the Liquid Helium Pool 168/II

See Abstract No. : 69/IV

A New Type of Helium Liquefier 169/II

United Kingdom. The helium liquefier described is of the 'external-work' type, making use of a bellows engine in place of the conventional cylinder and piston expansion engine. The bellows engine possesses a number of advantages: It does not require lubrication and since the 'cylinder' is gas-tight it can be placed in a vacuum enclosure for thermal insulation; moreover, since the mass is small, the expansion part of the system works with nearly perfect reversibility. The main difficulty anticipated in the development of this liquifier was fatigue failure of the metal bellows. A brass bellows of 4 cm. internal and 5.7 cm. external diameter, tested at the working pressure of about 5 atmospheres and employing a stroke of 2.7 cm., lasted for about 5×10^3 cycles at room temperature, 5×10^4 cycles at liquid air temperature and 5×10^5 cycles at liquid hydrogen temperature. A reduction of the stroke should increase the life of the bellows. The helium is supplied to the liquefier from a compressor at the rate of 13 m³ per hour (N.T.P.) at 20 atm.; 70% of this is reduced to 5.5 atmospheres by a regulating valve and is supplied to the bellows engine: 30% enters the Joule-Thompson system. Both gas supplies pass through a heat exchanger cooled by the outflowing gas, then through a liquid air bath at 80°K and then through the main heat exchanger. Next the 5.5 atm. gas passes through the bellows engine where its pressure falls to 1.3 atm. This gas is then used to cool the 20 atm. gas to 15°K before it undergoes Joule-Thompson expansion which results in its liquefaction. The whole liquefier is surrounded by a Dewar vessel containing liquid air and the portions beyond the liquid air bath are contained in an inner vacuum vessel. The bellows engine is normally run at 2 to 3 revolutions per second and the liquefier produces 0.6 litres of liquid helium per hour, consuming 1.4 litres of liquid air. The paper gives constructional details of the bellows engine and discusses steps which are being taken to improve the efficiency of the liquefier.

Sommaire: Description d'un liquéfacteur d'hélium employant un soufflet à la place du cylindre et du piston conventionnels.

Article by H. M. Long & F. E. Simon *Appl. Sci. Res.* **4A**, 1954 237-242

A Linde Helium Liquefier-Cryostat Combination Suitable for Operation Down to 1.3°K 170/II

United Kingdom. A helium liquefier and cryostat is described which is characterised by its inexpensive construction. Helium gas at 390 p.s.i. is led through a heat exchanger to a pre-cooling bath and charcoal cleaner in liquid nitrogen in a glass Dewar and is then passed, *via* the central tube of a concentric pair, externally lagged, to an adjacent concentric nest of Dewars, where it is then led, through a second heat exchanger coil, to a pumped liquid hydrogen bath. Thence, *via* a third heat exchanger, the high pressure helium is expanded, in an adjustable Joule-Thompson valve, into a liquid helium vessel, beneath which is attached a vacuum case, in which a calorimeter may be mounted as adiabatically as vacuum and support conduction arrangements permit. The unliquefied helium gas, leaving the Joule-Thompson valve at 6.5 p.s.i. is returned, *via* the

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outer tubing of the third heat exchanger, the second heat exchanger, the outer tube of the concentric pair to the top of the pre-cooling Dewar, and the outer tube of the first heat exchanger to an exhaust tube which may be connected to a pump for reducing the temperature of the liquid helium bath down to 1.35°K if required. The liquefier is initially pre-cooled by liquid nitrogen with exchange gas in the vacuum insulation space surrounding the liquid helium chamber. The liquid nitrogen is pumped to 65°K and the exchange gas evacuated. The liquid nitrogen is blown out and liquid hydrogen admitted to its Dewar, with exchange gas in the vacuum case again, and helium gas is circulated at low pressure. The liquid hydrogen is then pumped to its triple point and the vacuum case evacuated. The helium is then increased to full pressure and the Joule-Thompson valve is adjusted. Liquid helium collects after a further 2½ minutes and liquefaction proceeds at the rate of 800 c.c./hour. 3.5 l. of liquid hydrogen are required to cool the apparatus and produce 200 c.c. of liquid helium. This includes 1.4 l. which remain in the system. A series of radiation shields are mounted above the level of the liquid hydrogen and cooled by the evaporating gas. The evaporation rate of hydrogen rises from 120 to 300 c.c./hr. in about 1½ hour. The main helium vessel has a capacity of 250 c.c. and the evaporation rate of helium (unpumped) is 1 c.c. in 5 min. The experimental chamber is 7 cm. diameter × 9.5 cm. long. Total starting-up time is 63 min. to beginning of helium liquefaction. Two examples of operation of the cryostat are given, one using a calorimeter in the experimental chamber supported by a long coil of fine tubing from the bottom of the liquid helium bath and provided with constantan wire thermometer and heater. With 0.2°K temperature difference between calorimeter and cryostat the heat leak is 5 erg/sec. of which 1.5 erg/sec. is due to pump vibration.

Article by D. H. Parkinson *J. Sci. Instrum.* 31, May 1954 178-180

Sommaire: Description d'un liquéfacteur d'hélium de Linde qui a un rendement de 800 c.c./heure et une économie raisonnable dans l'emploi d'hydrogène liquide.

An Efficient Cryostat for Producing Temperature between 4° and 80°K: The Production of Liquid Hydrogen Targets Using Liquid Helium 171/II

See Abstract No.: 193/III

Vacuum Flasks of Unusual Design 172/II

United States. Small liquid oxygen containers developed recently at the National Bureau of Standards for the storage of supplies of breathing oxygen in military aircraft have introduced several new features in Dewar flask construction of potential use in the design of larger vessels. There are three types of the new containers, all cylindrical in shape. Their capacity is about 1.7 l. In order to obtain as long a heat conduction path as possible between the cold liquid and the surrounding atmosphere in a restricted space the neck of one of the three types of containers doubles back into the vessel and through the liquid, insulated from it by an extension of the vacuum space. Thus a neck of about 11-inch. length is obtained and gas and liquid connections of the vessel are at the bottom. The neck is made of Inconel tube of 0.25 inch inner diameter, the container is made of brass, and the vacuum applied is 0.5 μ Hg. Oxygen is lost by evaporation at the rate of 0.070 lb. of liquid oxygen per hour. The design of the second and third type of container fulfils an additional requirement in military flying, *i.e.* it can be inverted without affecting heat leak conditions. This is achieved by arranging for the inner chamber of the flask to be supported at the top and at the bottom by two identical neck drains. Whatever the position of the container the drain situated at the top serves as a gas vent and the drain situated at the bottom is employed for adding liquid. The evaporation rate of the two latter designs is 0.065 lb. per hour and 0.073 lb. per hour respectively.

Note by Anon *Lab. Practice* 3, August 1954 349-350

Sommaire: On donne des détails sur des petits vases à oxygène liquide récemment developpés par le Bureau National des Standards; ils pourraient être d'un intérêt futur dans le développement de grands vases Dewar

Simple Safety Device for Hg Diffusion Pumps 173/II

See Abstract No.: 136/II

A Universal Apparatus for High-Precision Pressure Regulation at Reduced Pressures 174/II

See Abstract No.: 145/II

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30 — EVAPORATION — 30

New Types of Interference Filters of Greater Areas and Higher Contrast 153/III

See Abstract No.: 235/I

Thickness Measurements of Thin Films by Multiple Beam Interferometry 154/III

United States. A brief discussion of Fizeau fringes and fringes of equal chromatic order is followed by data on the thickness measurement of thin films of silver, barium stearate, and collodion by means of fringes of equal chromatic order. Measurements were made on films ranging in thickness from about 100 to 2,000 Å with a probable accuracy of 15–30%. Mostly the films to be subjected to interferometric measurement were not formed directly on the microscope slide but were prepared on some other support using collodion as a substrate to facilitate the transfer to the microscope slide. In the course of the experiments the authors investigated whether measurements on a collodion film of the type mentioned would indicate any change as a result of the stripping operation required to facilitate the transfer. A microscope slide dipped in 1% collodion solution was drained, dried and cut in half lengthwise. One half was stripped and transferred to a second slide. Both parts were scratched in three corresponding planes and the thickness was measured, with the results shown in the table partly reproduced below.

Comparison of Stripped and Unstripped Films

Scratch	*Film Thickness* (Å)	
	Unstripped	*Stripped*
1	1310	1276
2	1446	1434
3	1567	1456

Sommaire: Des recherches ont été faites sur la technique d'interferomètrie pour son emploi dans les mesures d'épaisseur de couches de collodion, membranes de plastique etc.

Report by T. W. Bartlett & F. L. Ball *A.E.C. Rep. No.* K-1135 June 1954

Usefulness of the Polarimetric Method for the Determination of the Refractive Index and of the Thickness of one Thin Film on Glass or on Metal 155/III

See Abstract No.: 234/I

On Polarisation by Transmission with Particular Reference to Selenium Films in the Infra-Red 156/III

See Abstract No.: 233/I

Two-Layer Anti-Reflection Coatings for Glass in the Near Infra-Red 157/III

United States. Three types of two-layer anti-reflection coatings capable of producing zero reflectance on glass of index 1.51 are discussed. All three types consist of a high-index inner film and a low-index outer film and use the same low-index film material (MgF_2 with $n_1=1.38$) but various high-index coatings. The first design, consisting of two quarter-wave films, requires $n_2=1.7$ for producing zero reflectance on glass at 1μ wavelength. The reflectance minimum of such double quarter-wave coatings is extremely sensitive to thickness variation of the low-index film and very much less influenced by mismatching of the high-index coating. The second type of coating consists of a thin high-index ($t_2<\lambda/4$) and a thick low-index ($t_1>\lambda/4$) film. This combination can yield zero reflectance for all values of $n_2>1.7$. The third coating type uses one half-wave and one quarter-wave film. With coatings of this type two reflection minima which become zero for $n_2=1.9$ can be obtained. Experimental coatings of the three types were used for anti-reflecting glass in the near infra-red. The coatings were prepared by high vacuum evaporation of MgF_2, Si0, and $Ce0_2$.

(*Authors*)

Sommaire: Trois types de dépôts, formés de deux films Anti-Réflexion capables de produire une réflexion nulle sur verre d'indice 1.51, sont discutés.

Article by J. T. Cox G. Hass & R. F. Rowntree *Vacuum* 4, Oct. 1954 445-455

Structure and Texture of Evaporated Nickel Films Determined by the Electron Microscope and Electron Diffraction 158/III

See Abstract No.: 241/I

An Electron-Diffraction Examination of Thin Films of Lithium Fluoride and Copper Prepared by Vacuum Evaporation 159/III

United Kingdom. The investigation reported is concerned with stress systems in thin films of lithium fluoride and copper deposited on a cellulose substrate by evaporation at a pressure of 10^{-5} mm. Hg. The films were

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examined by electron diffraction. The ratio of the rings was measured with an accuracy of 1 part in 10^4 and it was thus possible to compare the spacings of the planes of different indices to a similar degree of accuracy. In previous work errors had been introduced by the charging up of the photographic plate by the undiffracted electron beam; the resulting electrostatic fields caused distortion of the diffraction pattern. In this investigation the undiffracted beam was intercepted by an earthed metal disc placed above the plate. A further improvement was the use of absolute measurements which necessitated a knowledge of the electron wavelength. This was determined to one part in 10^4 by measuring the accelerating potential with a special potentiometer devised by Rymer and Wright. According to the theory of elasticity a uniform hydrostatic pressure applied to a cubic crystal causes the same strain in all directions and consequently a uniform expansion or contraction of the diffraction pattern. Thus, such a pressure cannot be detected by relative measurement of the radii of the diffraction rings but only by absolute measurement of the radii. The measurements of the radii of the diffraction rings were used to deduce the interplanar spacings, and hence the stresses in the specimens. The theory of the effect of stress and specimen texture on the diffraction pattern is given. The results show that specimens of lithium fluoride have one principal axis of stress of magnitude $2p$ perpendicular to the specimen plane and the other two axes of magnitude p in the plane of the specimen. Such a stress system leads to the conclusion that the crystals have the form of cylinders with their axes normal to the specimen plane and that the force acting on the cylindrical crystal is surface-tension. The cylinders were found to have a diameter of about 100 Å and the surface tension is estimated to be of the order of 1,000 dyn/cm. The (100) crystal axis tended to lie along the axis of the cylinder. The investigation of copper films showed that these consist of crystallites in the form of discs, of a diameter of about 90 Å and a thickness of about 15 Å parallel to (111) planes. It was established that the stresses existing in these films are caused by superficial oxidation of the specimens as a whole.

Article by J. S. Halliday T. B. Rymer & K. H. R. Wright *Proc. Roy. Soc.* **225A**, Sept. 1954 548-563

Sommaire: Des couches fines de fluorure de lithium et de cuivre évaporés sous vide, ont été examinées avec un appareil à diffraction d'électrons employant des méthodes nouvelles, afin d'examiner la tension dans ces films.

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A High-Resolution Evaporated-Carbon Replica Technique for the Electron Microscope

160/III

See Abstract No.: 148/II

Apparatus and Process for Coating by Vapour Deposition

161/III

United States. In continuous-band coating it is desirable to have a large supply of the metal to be evaporated available within the coating chamber in order to coat large areas of the strip in one run. Further, it is desirable to minimise the heat incident on the strip by radiation. A device is described which fulfils both these requirements and is essentially designed to be operated with aluminium as the coating material. The plant consists of a conventional work chamber which can be evacuated to a few micron Hg. The strip to be coated is conveyed from one spool to another by conventional means and is exposed, on its passage, to the vapour. The evaporation source is designed to hold a large amount of aluminium at a temperature only just above its melting point. The crucible is preferably made from graphite and covered by a refractory plate. The cover is slotted to permit the upper edge of a plate situated in the centre of the crucible to project through it. This plate is preferably of a length equal to the width of the substrate to be coated and in a position rectangular to the direction of the motion of the substrate. Whereas the lower portion of the crucible is heated to just above the melting point temperature of the aluminium, the upper edge of the projecting plate is heated by a separate induction coil to 1,300°C or above. Like the walls of the crucible the central plate is preferably made from carbon or a mixture of carbon and graphite, but the surface of the latter is treated to form a carbide of a metal of group IVa or Va in the Periodic Table. Such a carbide surface can be obtained by dipping the plate, under vacuum, into a molten bath of aluminium and zirconium. As a result of such treatment, and assisted by the higher temperature at the upper edge, the component will act as a wick and the aluminium will climb to the top of the plate from which it evaporates and deposits on the substrate. A convenient rate of evaporation obtainable with this device gives approximately 0.7 g. per 30 sq. ft. of substrate area.

P. Godley 2nd & National Research Corp. *U.S. Pat.* 2,665,225

Sommaire: Description d'une source d'évaporation donnant un minimum de chaleur par radiation. Elle consiste en un élément recouvert de carbure de zirconium, fixé au centre d'un creuset en carbone, et agissant comme une mèche pour l'aluminium fondu.

Process for Depositing an Aluminium Film on a Substrate by Thermal Vaporisation

162/III

United States. Carbon (or graphite) crucibles, commonly used in vacuum evaporation processes employing aluminium, suffer from the disadvantage that at the required operational temperatures, *i.e.* at 1,200–1,300°C, the molten aluminium attacks the carbon and forms a carbide which has a limited solubility range and tends to crystallise out at the (cooler) evaporating surface, producing a floating scum. The presence of this scum diminishes the effective evaporation surface, and, in consequence, the rate of evaporation. Particularly in evaporation plant, where solid aluminium is fed to the molten bath, continuous scum formation may reach proportions where the evaporating process is seriously interfered with. The object of the present invention is to overcome this difficulty by introducing into the crucible a third material capable of forming, with the aluminium a carbide which is practically insoluble; suitable materials are: Hafnium, zirconium, titanium, tantalum, and vanadium, but the use of zirconium is preferred. The chosen element may be introduced into the graphite crucible prior to or during evaporation, either in its elemental form (by deposition or as powder or pellets) or as a reducible compound, though in general this is less effective with regard to high evaporation

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rates. In either case the reaction is to form an insoluble carbide which is heavier than aluminium and therefore sinks, forming effectively a lining to the crucible. At 0.5 mm. thickness, corresponding to an addition of some 2 g./sq. inch of the interior surface of the crucible, it is capable of protecting the crucible wall from attack by the molten aluminium. Alternatively, the chosen third material may be used to form the whole crucible. However, apart from the difficulties of forming and machining, the crucible would be expensive and more sensitive to heat shock.

Sommaire: L'emploi de creusets de carbone dans les appareils à évaporation d'aluminium sous vide, cause une formation de carbure d'aluminium qui est insoluble et produit une écume flottant sur la surface d'évaporation. L'addition de zirconium au bain prévient cette difficulté.

P. J. Clough
E. E. Chadsey Jr.
& National Research Corp.
U.S. Pat.
2,665,223

Printed Circuit Technique 163/III

See Abstract No.: 230/I

The Zinc Coating of Paper for Capacitors by Vacuum Evaporation 164/III

United Kingdom. The advantages are discussed of metallised foil and paper construction of capacitors. It is stated that the volume of a conventional capacitor may be reduced to $\frac{1}{3}$ by the use of metallised paper, and that this paper possesses valuable self-healing properties. The film on the paper must be continuous and thick enough to be a good electrical conductor, and must adhere well without penetrating. Both zinc and aluminium have the necessary resistance to atmospheric corrosion and may be evaporated in vacuum at high rates to condense as uniform conducting films. Zinc is preferred to aluminium since it has a much higher vapour pressure for a given temperature, and it may also be condensed at high residual gas pressures without deleterious effects. Although no comparative information on the performance of zinc and aluminium coated capacitors is available, it is suggested that zinc capacitors might have better self-healing properties, but that aluminium capacitors are more resistant to oxidation. In the zinc coating process here described, the metal is evaporated from an electrically heated crucible in a continuously pumped vacuum chamber. The paper to be coated is passed over the crucible and a thin uniform film of zinc is condensed on the surface. The factors governing the quantity of the coating are discussed. A highly volatile metal such as zinc requires either a very high vapour beam intensity or a well-cooled surface to obtain adequate condensation. This difficulty has been overcome by first evaporating a thin silver film on the paper which serves as a nucleating layer upon which the zinc atoms condense rapidly. Methods of preventing metal deposition on parts of the coating equipment and on the margin of the paper are mentioned: This is accomplished by coating these parts with a thin film of low-vapour pressure oil, such as silicone diffusion pump oil. Rolls of capacitor tissue normally contain about 8% by weight of water, which must be removed before a satisfactory coating can be obtained. Vacuum drying systems are described, including both refrigerated systems and systems using air-ballasted rotary pumps for the removal of the water-vapour. Finally a typical commercially available plant for the roll coating of capacitor tissue with zinc is described. Both zinc and silver sources are provided. The paper may be coated at rates up to 500 ft./min.

Sommaire: On discute la production de papiers pour capacités par les méthodes d'évaporation thermique ainsi que le revêtement de tels papiers avec du zinc.

Article by
L. Holland &
K. Hacking
Electronic Engng.
26, July 1954
296-301

Catalysis on Evaporated Metal Films. III. The Efficiency of Different Metals for the Reaction Between Ethane and Deuterium 165/III

See Abstract No.: 252/I

31 — CATHODIC SPUTTERING — 31

Cathode Sputtering in Glow Discharges 166/III

United Kingdom. There are three distinct aspects of the study of cathode sputtering in a glow discharge: (*a*) The atomic mechanism of the disintegration of the cathode, (*b*) the movement of the sputtered atoms through the gas, and (*c*) the condensation of the atoms on the surface. The present paper reports on a theoretical investigation of the second aspect, which is the most important as information on (*b*) is necessary for the proper understanding of (*a*) and (*c*). Previous theoretical treatments have made assumptions and approximations which restrict the range of application of the results, and big discrepancies have been observed between theory and practice. The treatment given here is based upon the geometry of a simple discharge tube with disc electrodes of a diameter equal to that of the tube. The partial pressure of the metal vapour is considered to be small compared with the pressure of the gas, and the mean free path of the metal atoms is considered to be small compared with the distance between the electrodes. A cosine law emission for the sputtered metal atoms is assumed, and their velocities are expected to obey approximately the Maxwell distribution law. The analysis does not follow simple diffusion theory, since it is not possible to specify proper boundary conditions, therefore the movement of the atoms is treated first as a scattering process. The concept of an 'initial' zone is introduced, where most of the metal atoms are assumed to undergo their first collisions. Thereafter, provided persistence is allowed for in the scattering process, the metal atoms diffuse through the gas. Expressions are derived for the total amount of metal leaving the cathode which does not diffuse back, and for the density of the wall deposit.

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Typical axial density distributions are plotted for aluminium atoms diffusing through neon under five different sets of conditions. Some agreement was obtained with the experimental results of other workers, but the need was shown for more information on experiments made in a simple apparatus of the type described. It was concluded that the theoretical complications were so great that experiments carried out in a glow discharge were unlikely ever to yield precise information about the atomic mechanism of cathodic sputtering.

Sommaire: Les différents facteurs impliqués dans le phénomène de projection cathodique ont été étudiés théoriquement, et on en conclue qu'il est peu probable qu'une théorie donnant des conclusions exactes à partir des données expérimentales, puisse être tirée dans l'avenir, du fait de la compléxité des phénomènes y prenant part.

Article by G. Ecker & K. G. Emeleus *Proc. Phys. Soc.* **67B**, July 1954 546-552

Light Absorption and Electrical Properties of CdO Layers with an Imperfect Lattice

167/III

Germany. The electrical properties of CdO layers produced by sputtering have been investigated by Lappe (*Z. Phys.* **137**, 1954, 380). It was noticed that the colour of the CdO layer was different whenever the conditions under which the layer was prepared had changed. It was decided therefore to follow up Lappe's electrical measurements by a series of optical absorption measurements on the same layers. The optical measurements were carried out in the range 450–700 mμ at room temperature on layers heat-treated previously at various temperatures up to 400°C and deposited originally in various gas mixtures (air, 3% oxygen–97% nitrogen and 21% oxygen–78% argon). It was found that the values of the extinction constant E are shifted in the direction of lower wavelength as the temperature employed in the heat treatment of the layers was increased. Thus, the shift of the value of the extinction constant E at a given wavelength λ_0 serves as a useful yardstick for the assessment of the changes obtained by different treatment of the layers. An analysis of the optical measurements obtained by this procedure appears to give somewhat erratic results but a comparison of the values of the extinction constant at λ_0 and electron concentration obtained in Lappe's investigations established a close relationship. As the electron concentration increases the value of the extinction constant is reduced and *vice versa*. Further research work is required to give a proper interpretation of these results.

Sommaire: Des mesures d'absorption optique faites sur des couches de CdO, examinées auparavant pour leurs propriétes électriques, permettent d'établir une relation étroite entre les valeurs de la constante d'extinction et la concentration d'électrons.

Article by H. Dunstädter *Z. Phys.* **137**, No. 3, 1954 383-385

Cathodic Vacuum Etching of Uranium

168/III

United States. The study of the surface structure of uranium in light and electron microscopes is handicapped by the limitations of the conventional specimen preparation methods. Uranium specimens prepared by such methods are liable to rapid oxidation. A novel method of preparing uranium specimens, overcoming these difficulties, provides for the etching of the uranium sample by sputtering in vacuum. It was found that specimens treated in this manner could be exposed to air at room temperature for a month without deterioration. The process is carried out in a 5 l. sphere containing an earthed aluminium anode and a zirconium cathode 2 inch. in diameter. The zirconium cathode, which was chosen because of its low sputtering rate is sealed to glass tubing through which a coolant can be circulated. Etching is effected in krypton gas, which permits relatively intense bombardment, introduced into the chamber through a controlled leak which, in conjunction with the evacuating equipment, maintains the pressure at 75μ Hg. The specimens, polished mechanically before treatment, are subjected to sputtering for about one hour at 3,000V and at a current density of about 3 mA/sq. inch of the cathode surface. Etching is preceded by a cleaning treatment lasting about ten minutes in order to eliminate residual oxides, and other contaminants, from the specimen surface. When the specimen is seen to be etched to a suitable extent, the pressure is raised to atmospheric by introducing pure helium and the sample is removed from the chamber. A special method for the preparation of effective replicas of etched uranium surfaces involving the use of Faxfilm is described. For examination in the light microscope, softened Faxfilm ribbon is placed on the surface of the specimen, allowed to dry, and then stripped and shadowed with aluminium. For electron micrography, two-step replicas are obtained by shadowing the Faxfilm replicas with a gold-manganin alloy, evaporating silicon monoxide at normal incidence on to the gold-manganin layer and finally stripping the compound replica from the specimen by dissolving the Faxfilm base in acetone. Specimens were prepared in this way from uranium samples which had been rolled in the α temperature range, β–heat treated and γ–extruded. Electron micrographs obtained from these specimens are reproduced.

Sommaire: Des détails sont donnés sur une méthode de préparation de spécimens d'uranium devant être éxaminés au microscope électronique; la surface du specimen est traitée par projection cathodique afin d'obtenir un effet de gravure.

Report by T. K. Bierlein *A.E.C. Rep. No.*: HW-32676 August 1954

Cathodic Vacuum Etching

169/III

United States. The merits of the cathodic vacuum etching method are briefly discussed and the history of the process is reviewed. This is followed by a detailed description of a newly developed cathodic vacuum etching plant which facilitates the satisfactory etching of a variety of materials, for the purposes of metallographic study, in periods ranging from 1 to 3 minutes as compared with 1 to 3 hours required in previous techniques. Essentially the plant consists of a 12–inch bell jar resting on a base plate which is fitted with connections to the ionisation gauge, the pumping unit and the argon supply. The pumping unit consists of a diffusion pump coupled with a mechanical rotary pump. During the etching period the pressure in the bell jar is maintained at 50μ Hg. The cathode is suspended from the top of the bell jar and consists of a face plate 1.125 inch. in

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diameter to which the sample is clamped prior to treatment. The material of the cathode is zirconium. The anode is supported by the base plate and consists of a $2\frac{3}{4}$–inch aluminium disc 4 inch. away from the cathode. Both electrodes are surrounded by a glass envelope which forms in practice an isolated glow cell within the vacuum chamber. A coolant tray is fitted to the terminal of the cathode outside the evacuated chamber and is filled with dry ice and trichloroethylene, acting as a heat sink during operation and thus facilitating high current densities on the cathode without excessively heating the sample. The high voltage source is a 5,000 V, 50 mA, full-wave rectified d.c. power supply with a positive ground and a 60,000 Ω current-limiting resistor. Even in runs lasting 6 minutes the temperature of the sample never exceeded 230°C. Details of the operation are given. A complete cycle takes about 20 minutes. Results obtained with the plant are shown in illustrations. All kinds of metals have been etched with success, including uranium. The only alloy which proved difficult was a 70–30 silver-cadmium alloy which etched so fast that the depth of the etch could not be controlled. Some complex materials such as graphitic Stellite, chromium carbide and ceramet (70% chromium, 30% alumina) can also be etched with success but the process takes longer. The zirconium fittings incorporated in the plant proved unsuitable in the case of etching pure aluminium or aluminium alloys. In that case noticeable re-deposition of material on the etched surface took place. Similarly zirconium could not be etched on aluminium fittings without showing a corresponding phenomenon. This difficulty was overcome by employing cathode fittings made of the same type of metal as the sample.

Article by T. R. Padden & F. M. Cain Jr. *Metal Progr.* July 1954

Sommaire: Description d'un nouvel appareil de gravure sous vide qui ramène les temps de gravure d'environ 1 à 3 minutes comparés avec les temps de 1 à 3 heures nécessaires avec les techniques habituelles.

33 — IMPREGNATION — 33

Impregnation of Small Friable Objects

170/III

United Kingdom. Reference is made to a note contributed by a member of the Department of Egyptology, London University, to the Museums Journal describing a simple vacuum method of impregnating small friable objects for the purposes of strengthening. Normally the objects are sprayed with polyvinyl acetate solution. This produces a glossy surface which can be removed by washing with toluene, but washing weakens the surface of the object. Total immersion of the object in polyvinyl acetate, dissolved in toluene at atmospheric pressure, gives better results but the best results are obtained by effecting the immersion in a chamber such as a vacuum desiccator which can be evacuated with a water jet pump for the purpose.

Note by Anon. *Nature* 174, 25.9.1954 587

Sommaire: Description d'une méthode simple d'imprégnation par le vide de petits objets friables, afin de les re-enforcer pour les exposer dans les musées.

36 — DRYING — 36

Developments in Meat Dehydration in the United Kingdom

171/III

United Kingdom. A brief description is given of the organisation of the Ministry of Food Research Establishment experimental factory at Aberdeen as an introduction to the report on the work carried out in that establishment on meat dehydration. The ideal characteristics of dehydrated food are given as follows: (*a*) Rapid reconstitution, (*b*) capability of replacing the original material in the preparation of any dish, (*c*) retention of colour, flavour, texture and nutritive value, (*d*) long storage life without refrigeration and (*e*) high bulk density. No method of dehydrating meat has yet been developed to fulfil all five conditions. A moderately successful method of dehydrating meat, *i.e.* the hot air drying method, was introduced in 1941. Hot air-dried minced cooked beef and mutton were produced on a commercial scale in the war and were highly acceptable. But the success of the method is limited to meat in the form of mince. Where cubed or sliced meat is to be dehydrated a vacuum process must be used. The main advantages of vacuum treatment are reduced risk of deterioration due to oxidation, and improved qualities of flavour and texture, because the processing temperature is reduced. The best vacuum method is freeze drying which gives a product closely approaching the ideal but having a low bulk density. This process is being actively investigated by Flosdorf in America but is rather expensive. The author continues by giving a detailed description of the method, and its results, of vacuum drying meat in accordance with the procedure outlined in British Patent 603, 970 (Aktieselskapet Atlas, Copenhagen, Denmark). Essentially the method, known as the vacuum contact–plate process, provides for the tissue to be dehydrated by heating in a vacuum between moveable hot plates. A large vacuum chamber contains a rack of 24 water-heated shelves, 8 × 4 ft., which hold the meat. The shelves are internally divided into a labyrinth to equalise the temperature across the shelf area during drying. The plates can be closed together by hydraulic pressure and the material to be processed is sandwiched between an aluminium tray and a lid placed between a pair of the heating plates. Each tray can carry 2 lb. of meat per square foot, *i.e.* one charge is 10 cwt. After charging and closing the cabinet, it is evacuated to an initial operational pressure of 10 mm. Hg, and during this period the temperature of the circulating water is about 100°C. As the drying proceeds the pressure is gradually reduced to about 3 mm. Hg and the temperature of the circulating water is gradually reduced to

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about 60°C. The water vapour evolved is removed from the cabinet by steam vacuum augmentors situated at the top of the cabinet and connected to a jet condenser. Permanent gases in the condenser are removed by twin water ejectors. By this method a charge of about 740 lb. of raw stewing beef chunks (approximately 1 inch cubes) can be dried down to a moisture content of 3% in approximately 8 hours. The conditions during drying are probably unfavourable to bacterial growth though the meat is unsterilised. The product can be reconstituted by soaking in water for about 1–2 hours. Some blood is lost to the water and, due to denaturation of the protein during drying, it resorbs water less easily. Therefore it tends to be tougher and its flavour is weak. The product is unsuitable for frying but it can be cooked and is acceptable in casseroles, pies and pot roasts. Packaging in cans is preferred, but laminated film pouches can be used if they have low moisture permeability. This can be ensured by including a 0.0007 inch thick aluminium foil.

Sommaire: Description de l'organisation du "Ministry of Food Research Establishment" d'Aberdeen; on donne un rapport détaillé des expériences faites sur la déshydratation de la viande par le procédé à plaque de contact, sous vide.

Article by E. J. Rolfe *Food Trade Rev.* August 1954 12-15

Powder Makes A–1 Apple Juice 172/III

United States. A powdered apple juice product of bulk density 0.9 g./ml. and about 2.7% moisture content has been produced by vacuum drying. The powder may be reconstituted in 1½ minutes with five times its bulk volume of cold water. Drying is carried out in an apparatus similar to that described by Eskew *et al.*, (U.S.D.A. Report No. AIC–315, August 1951), concentrate of 80° Brix being spread on pans nesting on water-heated shelving in a vacuum oven containing a water-cooled condenser. The water removed is at first trapped on the condenser, then, as drying proceeds, condensate is drained off and the water vapour removed directly through the oil-sealed rotary vacuum pump which is equipped with an oil clarifier. The product foams at the start of drying but ends as a molten mass which becomes brittle and glass-like when cooled. The vacuum falls to about 0.2 inch. Hg at the end of the run. A dried bottle, containing tough sift-proof moisture-permeable paper envelopes filled with calcium oxide, is used for storing the dried material till it is ground in a hammer mill to pass a 10–mesh sieve. Grinding is possible as long as the material is cooler than 90°F and has a moisture content below 3%. A relative humidity of 15% is maintained during all processing steps. Slow mill speeds and coarse screens are used to get the minimum of fines to prevent caking of the ground material. The product is vacuum-packed into 4 oz. cans which contain another calcium oxide desiccant bag to maintain the low moisture content during storage. Three types of material were investigated for drying, all based on a blend of fruit juices which had been stripped of essence, depectinised, clarified and concentrated to 83° Brix. This concentrate was dried (*a*) as it stood, and (*b*) with a sucrose content equal in weight to the apple solids, with sufficient citric acid and essence to maintain the ratio of these to the total solids, and (*c*) as in case of (*b*) except that the sucrose was replaced by dextrose. These three concentrates were dried and reconstituted for a trained taste panel to sample: (*a*) Had an excellent fresh apple flavour. The flavour of (*b*) was virtually the same and (*c*) was decidedly deficient in apple flavour. As the cost of (*b*) was less than that of (*a*), its flavour at least as good and its storage properties probably improved by the sucrose addition, (*b*) was used for the pilot drying programme. A study of the storage properties of the dried concentrate is now being undertaken.

Sommaire: Déscription d'une méthode de production de poudre de jus de pomme d'une densité de 0.9 gramme par millilitre et d'environ 2.7% d'humidité; ce jus peut être reconstitué en 1½ minute en faisant dissoudre la poudre dans 5 fois son volume d'eau froide.

Article by H. I. Sonnamon V. A. Turkot R. K. Eskew & G. W. Macpherson Phillips *Food Engng.* **26**, July 1954 78-79

37 — METALLURGICAL PROCESSES — 37

Metallurgical Trends of Interest to Chemical Engineers 173/III

See Abstract No.: 215/I

Preparation of High-Purity Lithium Metal by Vacuum Distillation 174/III

United States. In order to investigate the physical properties of pure, or nearly pure, isotopes and their compounds of lithium, small quantities of highly purified lithium were required. A laboratory-scale vacuum distillation method, for the purification of lithium obtained by the reduction of the compounds of the metal by conventional means, is described. The apparatus was of the pot-still type, the pot being formed by a piece of stainless steel tubing of 1⅞ inch outer diameter and 5/32 inch wall thickness. The pot was supported by a piece of stainless steel tubing 1½ inch in diameter and of a wall thickness of 1/16 of an inch. The supporting tube rested on a stainless steel plate and was in communication with the pot by means of a tube projecting through the bottom of the pot. The pot, which was covered by a removable lid, was surrounded by a nichrome ribbon heater element which facilitated heating the lithium charge to about 500°C. The vapours, passing through the communicating tube, condensed on the walls of the supporting tube. Impurities condensed at different levels from pure lithium. The whole assembly, including a hood made from kaolin firebrick supported by a separate structure from the base plate, was placed in a chamber made from black iron which could be evacuated. A 5 g. charge with an initial impurity content of 2 to 3%, treated at 500°C for about four hours at an operational pressure of 0.05

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micron Hg, gave about 3 g. of 99.5+% pure lithium. Strontium was the only impurity presenting difficulties in separation. Detailed results are given in a table partly reproduced below.

Constituent	*Run No. 1 (Electrolytically Reduced Metal)*		*Run No. 3 (Thermally Reduced Metal)*	
	Initial %	*Final* %	*Initial* %	*Final* %
Al	>1	T‡	ND	ND
Ba	ND§	T	0.03	T
Ca	0.05	T	0.5	T
Mg	T	T	0.3	T
Na	0.5	<0.05	0.1	≃0.05
Si	0.1	T	ND	ND
Sr	ND	ND	>1	1

‡ T = trace (<0.02%) § ND = not detected

Article by P. S. Baker F. R. Duncan & H. B. Greene *Science* 118, Dec. 1953 778-780

O *Sommaire*: On donne des détails sur un appareil de distillation sous vide pour purifier le lithium au taux de 5 grammes par charge.

Method and Apparatus for Producing Ions 175/III

See Abstract No.: 79/IV

Ion Source 176/III

See Abstract No.: 78/IV

Improvements i.o.r.t. the Vacuum Fusion and Treatment of Metals and Alloys and Apparatus Therefor 177/III

Liechtenstein. A method is described for the preparation of metals or alloys of high purity, *e.g.* iron and steel with a known low carbon and oxygen content. Melting iron at atmospheric pressure will neither achieve complete decarbonisation nor complete deoxidation, due to the formation of a carbon monoxide atmosphere above the melt which will cause the corresponding reaction to stop as soon as equilibrium is obtained between the melt and the gas phase above it. This difficulty can be overcome by melting under vacuum, but unless the carbon and oxygen content of the material prior to treatment is known, the results are not satisfactory. Therefore, in order to obtain iron of highest purity, a procedure is employed whereby excess oxygen is admitted to the evacuated furnace during the melting down process of the material, followed by treatment under vacuum to cause dissociation of the iron oxides. The method can be accelerated by admitting hydrogen to the furnace but this involves special treatment to remove the hydrogen afterwards. In the present method high purity iron of known and predetermined oxygen and carbon content is obtained by melting down the material, admitting oxygen to the evacuated furnace, and dipping a known quantity of the degassifying substance, *i.e.* graphite or carbon, into the melt for the period required to complete the reaction to the desired degree. The progress, or alternatively the completion of the reaction, can be followed either by measuring the pressure in the vacuum furnace or alternatively by visual observation through a window in the lid of the furnace. In order to obtain accurate results it is desirable to use induction heating which agitates as well as heats the metal. Details of the mechanism which facilitates the manipulation of the body of the degassifying substance are given. The method can be applied on an industrial scale and the processing time required is appreciably shorter than that of methods developed in the past for the purpose.

O

Sommaire: Description d'une méthode pour la préparation de métaux ou alliages ayant une petite quantité prédéterminée de carbone et d'oxygène.

A. Vogt *Brit. Pat.* 715,187

Vacuum Heat-Treatment 178/III

Canada. A laboratory-size vertical tube furnace is described, designed for annealing polished specimens of alloys of pure titanium and zirconium without tarnishing the surface, to facilitate metallographic studies. The tube is either of mullite with ends plated with copper over silver to facilitate soldering or of Inconel X, and has a diameter of $1\frac{3}{4}$ inch. The heat is supplied by six $\frac{1}{2}$-inch diameter globars. Conventional methods of insulation are employed. A water jacket is soldered to each end of the mullite tube. The lower water jacket is sealed to a vacuum-coupling chamber which contains a small receptacle filled with Octoil oil. An opening in the top of the coupling chamber in the axis of the mullite tube is normally closed by a moveable small radiation shield. The specimen to be treated is suspended in the centre of the furnace by means of thin tungsten or molybdenum wire. For quenching purposes the suspension wire is fused electrically and the released specimen drops into the oil-filled receptacle in the coupling chamber. The vacuum-coupling chamber is cast from an alloy with the composition of 85% copper, 5% tin, 5% zinc and 5% lead. This showed satisfactory leak tightness when evacuated down to pressures of 5×10^{-6} mm. Hg. The furnace was evacuated by a 4–inch diffusion pump unit.

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The power supply was obtained from a 7 kW Variac with an output from 0–270 kV. At 1.6 kW input a temperature of 1,100°C was obtained in the mullite tube. The temperature could be controlled within $\pm\frac{1}{2}$°C at all temperature levels up to 1,100°C. Test runs on very pure iodide titanium specimens treated for periods up to 5 hours at temperatures of 500, 800, 900 and 1,000°C produced no change in the hardness of the material (95 V.H.N.). These results indicate satisfactory vacuum conditions in the furnace as above 880°C titanium is highly susceptible to contamination by common gases.

Sommaire: On donne des détails sur un four à vide vertical pour laboratoire, developpé pour le recuit de specimens polis, de titanium pur ou de zirconium, sans les ternir, ce qui facilite les études métallographiques.

Article by W. D. Bennett *Metal Industr.* 26.3.1954 245-246

Linings for Induction Furnaces

179/III

United States. Refractory materials are limited, commercially, to granular aggregates of crystalline alumina, crystalline magnesia, silica and zircon. Zirconia, beryllia and thoria are too expensive, and chromium refractories suffer from shrinkage during use. Alumina and magnesia mixtures and zircon are the most satisfactory commercial linings. Details on the performance of such linings are given in a table, partly reproduced below.

Relative Service Figures of Linings

Type of Alloy Melted	*Type of Lining*	*Size of Unit, lb.*	*Life in Heats*	*Type of Operation*	*Pouring Temperature*	*Remarks*
Stabilised stainless steel	Magnesia-alumina, 60–40	2,000	60 to 170	Intermittent	3,000 plus	Some patching done
Ni–Cr and Mo–V–Mn steel	Magnesia-alumina, 60–40	1,000	30 to 40	Intermittent	3,000 plus	No patching
Heat resisting alloys	Magnesia-alumina, 70–30	1,000	15 to 20	Intermittent	3,000 plus	Some patching
Low-carbon steel	Zircon	3,000	30 to 50	Continuous	2,950	No patching

Procedures for lining furnaces are described with many practical details. Patching methods are also discussed. Extensive patching is shown to be uneconomical and the patching of bottom linings is not advised. In the case of 24–hr. operation it is normal practice to double the number of units required so that the furnaces can be alternated day to day and maintenance work can be carried out on those which are down. Excess slag may be removed on those that are down, but only the warts and adhesions should be removed. Washes of high alumina cements will fill-in hairline cracks in graphite crucibles. Other crucibles may be repaired with washes made from cements of the same material as the crucible. The physical properties of commercial refractories are discussed with reference to equilibrium diagrams and graphs of thermal conductivity, linear thermal expansion and electrical resistivity. The relation between particle shape and packing density in refractory aggregates is indicated.

Sommaire: On discute l'emploi et l'entretien de differents revêtements pour four qui peuvent être obtenus commerciallement; on donne des informations et les propriétés des minéraux employés.

Article by H. E. White *Metal Progr.* September 1954 99-106

38 — DISTILLATION — 38

Apparatus for Molecular Distillation

180/III

See Abstract No.: 222/I

A Micromolecular Still

181/III

United States. The alembic pot method, as used for analysis of fatty methyl esters for monomer, dimer, trimer and high polymers, is unsatisfactory for the analysis of methyl eleostearate or where only small quantities of very pure material are available. The present method is a modification of that devised by Booy and Waterman. About 0.5 g. of the sample is dispersed on a small bale of glass wool suspended from a quartz helix with a sensitivity of 2.92 cm. displacement per g. load. A cross-wire below the helix is read against a scale mounted on the outside wall of the containing vessel. The latter is a highly evacuated glass tube of 3 cm. inner diameter with ground joints at either end. The distillation area is surrounded by an aluminium block which can be heated to thermostatically controlled temperatures. The lower part of the tube is cooled by a jet of air and serves as the condenser. A T–junction is joined to the lower end of the tube, connecting (*a*) to the vacuum pump unit and (*b*) to an ampoule or a trap. The trap model is employed for routine analysis not requiring the isolation of monomer and dimer. The ampoule can be sealed off under vacuum without interrupting the distillation. If a pure dimer fraction is required the apparatus must be disconnected after the monomer has distilled to clean the vessel of any monomer condensate before distillation is continued. A mercury diffusion pump unit is employed for evacuation. The operational pressure is about 1.5 μ Hg. The operating temperature

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is of the order of 250°C. Details of the operational procedure are given. Test runs on polymerised methyl esters, analysed previously by the alembic pot method, gave results agreeing within 1–3%, see table partly reproduced below.

Polymerised methyl ester		% *Monomer*	% *Dimer*	% *Trimer*
10, 12-t, t-linoleate 3 hours at 270°C	Previous method	37.8	58.8	3.1
	Present method	38.9	60.2	1.9
Linolenate 6 hours at 290°C	Previous method	57.4	31.9	9.9
	Present method	61.9	29.9	8.2

Sommaire: Description d'une micro-méthode de distillation moléculaire basée sur la méthode de Booy et Waterman, qui facilite l'analyse de méthyl éléostéarates.

Article by R. F. Paschke J. R. Kerns & D. H. Wheeler
J. Amer. Oil Chem. Soc.
31, No. 1 1954
5-7

Separation of Fluoranthene and Chrysene by Molecular Distillation

182/III

United States. The separation of closely related polynuclear hydrocarbons is difficult. Crystallisation methods may lead to the formation of solid solutions and vacuum fractional distillation methods may present operational difficulties. The present article gives the details of a successful procedure which essentially consists of dissolving the mixture of polynuclear carbons in a suitable polyglycol mixture and treating the solution in a cyclic molecular still. The method is based on Hickman's 'analytical distillation' whereby the substance to be investigated is dissolved in a carrier oil in preparation for the separation. The degree of elimination of the solute is a function of its concentration and its distillability. The latter rises with the temperature and the former can be determined experimentally by constructing the elimination curve showing the temperature at which the distillate contains the maximum concentration of solute. Glycols are soluble in water facilitating the recovery of hydrocarbon materials, and are available commercially in a wide range of molecular weights, and therefore have been considered suitable as a carrier material in the experiments described. The materials investigated were technical grade fluoranthene and technical grade chrysene both available from Reilly Tar and Chemical Corp. Details of the experimental procedure are given. Five experiments are reported. The first was concerned with the determination of the elimination curve at a pressure of about 30–35 μ Hg of a solution of 12 g. of fluoranthene in 750 g. of a mixture of glycols (polyethylene glycol 200, 300 and 400, and tetraethylene glycol). The second experiment was concerned with the determination of the elimination curve of chrysene at approximately 100 μ Hg pressure employing a solution 0.3 g. of chrysene and 627 g. of mixed glycols of a similar composition. The results of both these experiments are shown in tabular form and in graphs and indicate that the difference of elimination temperatures (fluoranthene: 110°C and chrysene: 150°C) was great enough to justify attempts at separation of the two hydrocarbons at low pressure. The remaining three experiments are concerned with the separation of a mixture of fluoranthene and chrysene employing a solvent containing equal parts of tetra-

Molecular Distillation of Mixture of Fluoranthene and Chrysene (Experiment 4)

Charge: Tetraethylene glycol	386.9	grams
Polyethylene glycol (160°–185° at 1–10μ)	386.9	,,
Fluoranthene	1.1029	,,
Chrysene	0.2917	,,
	775.1946	,,

Fraction	*Rotor Temp. °C*	*Pressure, μ Hg*	*Fraction Weight, Grams*	*Fluoranthene*		*Chrysene*		*Fluoranthene (Glycol-Free Basis), %*
				Concn. $\times 10^2$ %	*Weight, mg.*	*Concn. $\times 10^3$ %*	*Weight, mg.*	
1	100	17.0	64.1	34.44	220.76	17.52	11.23	95.15
2	100	15.0	75.8	30.08	228.01	23.94	18.15	92.62
3	100	16.0	57.6	28.23	162.61	26.31	15.16	91.47
4	110	1.2	87.4	26.48	231.44	47.80	41.78	84.71
5	120	0.4	51.1	22.93	117.17	86.56	44.23	72.60
6	130	0.6	35.4	19.51	69.07	159.02	56.29	55.10
7	140	2.6	19.0	11.36	21.58	183.70	34.90	38.21
8	150	0.8	18.4	5.97	10.99	137.27	25.26	30.32
9	160	1.7	20.9	2.90	8.62	62.23	18.61	31.78*
10	170		39.2	1.18	4.63	20.60	8.80	36.43*
11	180	10.0	62.5	0.64	4.00	5.73	3.58	
12	190	4.0	71.5	0.40	2.86	1.87	1.34	
13	200	2.5	88.6	0.39	3.46	0.94	0.83	
14	210	3.5	32.9	0.84	2.76	0.49	1.61	
Residue			25.3					
Loss			16.6					
Total			775.3		1088.01		281.05	

* Values after fraction 10 are inaccurate because of interfering absorbance of polyglycol.

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ethylene glycol and a polyethylene glycol fraction boiling at 160°C-185°C in the pressure range of 1 to 10 μ Hg. Detailed results of experiment four are reproduced on the previous page. While a satisfactory amount of fluoranthene was recovered from the mixture in a fairly pure condition, repeated distillation of fractions 7-14 (experiment number five) was required to secure relatively pure chrysene.

Sommaire: Détails sur une méthode de séparation des hydrocarbones poly-nucléaires semblables, basée sur une modification de la technique de distillation moléculaire developpée par Hickman et appellée distillation analytique.

Article by A. Cantoni J. Feldman & M. Orchin *Analyt. Chem.* **26**, Aug. 1954 1374-1377

Evaporation of Resting Water

183/III

United States. Previous measurements on the evaporation coefficient of water have given values of 1%–4% of theoretical values. Various hypotheses have been advanced for this discrepancy. The authors have made an exploratory survey of the conditions by examining the torpidity phenomena occurring on the surface of ordinary cold water under resting conditions. The term ordinary water is to be interpreted as all types of water excluding cloud or falling rain water. Water of a temperature near freezing point has a saturation pressure of 5 mm. Hg. In order to obtain schizoid patterns, therefore, it is necessary to have a vacuum of 3–4 mm. at least. If evaporation took place under these conditions without any obstruction, freezing would be instantaneous, but if there was a substantial obstruction freezing may be delayed to afford brief observations. Accordingly a pot still was designed fitted with a condenser, operated with trichlorethylene and/or dry ice, connected to a mechanical oil pump. A radiant heater of about 750 W was provided and arrangements were made to take photographic records of the surface formation if and when required. After cleaning the apparatus with acetone and distilled water, the flask of the still was half-filled with distilled water and the pump was switched on. For the first half-hour evaporation proceeded with bumping. When the pressure had reached 2 mm. one major crater appeared on the water surrounded by a number of small craters. Shortly afterwards the surface iced up. The apparatus was left standing overnight without breaking the vacuum and after sixteen hours the experiment was repeated but with different results. Instead of the small craters two or three large craters appeared which deepened rapidly and explosively, in one case leading to breakage of the still. Water samples left in the still for two days refused to perform at all. However, if the old charge was replaced by double-distilled water and if the apparatus was washed in between thoroughly with acid, alkali and water and thoroughly steamed, ice formed before any crater appeared and the pressure at no time decreased below 3.5 mm. The authors conclude from this investigation that water in general contains minute quantities of chemical impurity which either originate from the earth's surface or from glass containers. It appears that these impurities are not removed in the temperature range up to 100°C. Suggestions that silica is the cause of the schizoid behaviour of the water surface could not be finally established in the experiments reported here. In conclusion, the authors point out that the high degree of torpidity exhibited at temperatures near freezing point need not necessarily be present at elevated temperatures.

Sommaire: Des recherches sur les phénomènes de torpidité d'eau froide ordinaire au repos, prouvent que la surface de l'eau présente une résistance inusitée a l'évaporation. On discute les causes de ce comportement.

Article by K. C. D. Hickman & W. A. Torpey *Industr. Engng. Chem.* **46**, July 1954 1446-1450

Maximum Evaporation Coefficient of Water

184/III

United States. Previous investigations into the evaporation and condensation processes of water have led to the conclusion that not more than one in twenty-five molecules of water approaching the surface from either side merges with the interfacial layer. Generally they have involved the measurement of evaporation into a foreign gas (air) or into near-saturated vapour. These methods have been used in order to avoid the great loss of heat, and consequent freezing, if a vacuum method were used in the experiments. The author reports on a vacuum method which largely overcomes this effect. The apparatus employed in the investigation is a modified flowing-stream tensimeter. Essentially the instrument provides for a fast moving stream of water continuously circulated by an impeller to issue from a jet and to enter immediately the orifice of another. Both orifices are in a highly evacuated chamber. The gap is 1.2 cm. long, and the time the water stream is exposed to the vacuum is of the order of 0.001 second, thus minimising the cooling effect. The vapour given off *en route* from one orifice to the other was collected in traps. Proper timing of each run and measurement of the quantity of distillate yields the information required to calculate the evaporation coefficient ε=(quantity of distillate collected)/(maximum quantity calculated). The value observed in the experiments indicate an evaporation coefficient for a clean, new water surface of not less than 0.25, which under certain assumptions may well reach unity. Detailed results are given in a table.

Sommaire: Le coefficient d'évaporation d'une surface d'eau est déterminé par mesures sous vide, sans encourir le danger de gèle a environ 0.25–1.0 comparés avec 0.01–0.04 rapportés auparavant.

Article by K. C. D. Hickman *Industr. Engng. Chem.* **46**, July 1954 1442-1445

39 — MISCELLANEOUS PROCESSES — 39

New Developments in Vacuum Testing

185/III

See Abstract No.: 224/I

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Manipulation of Radioactive Gases in High Vacuum Apparatus

186/III

United Kingdom. The properties of radioactive isotopic gases, in particular tritium, krypton–85 and xenon–133, are described and an estimation of the radioactivity to be expected is given. The equipment available in A.E.R.E. Harwell for handling these gases is described in great detail with particular reference to the provisions made for protection against health hazards. The plant is made entirely of glass, the safe thickness of which can be calculated. Details are given of the vacuum system employed. Special manipulations described in the article cover the production and purification of tritium, tritiated water preparation, the preparation of tritium-zirconium targets, and the purification of krypton–85 and xenon–133. Besides their uses as research tools, the three nuclides are being increasingly used in industry. Among the applications mentioned are the uses of tritium for stabilizing the break-down voltage in thermionic valves, the advantages of krypton–85 when employed for the elimination of static charges and the uses of krypton–85 and xenon–133 for air-flow indication.

Sommaire: Les proprietes de gaz isotopiques radioactifs, en particulier le tritium, krypton–85 et le xenon–133 sont données, ainsi qu'une estimation de la radioactivite devant en résulter. Description est faite, en grand détail, de l'équipement disponible auf A.E.R.E. d'Harwell pour manipuler des gaz.

Article by E. J. Wilson *Vacuum* **4**, July 1954 303-325

The Production of Radio Isotopes

187/III

Holland. The methods used in the production of radioactive isotopes in the Philips synchrocyclotron at Amsterdam, employing a beam density of 30 μA, are discussed. Radio isotopes are obtained in nuclear reactions by exposing stable nuclei to a beam of fast light nuclei such as γ–particles, deuterons and neutrons. The former two are produced in cyclotrons, the latter normally in nuclear reactors. Most products obtained by bombardment with neutrons can also be produced by direct bombardment with deuterons which is the preferred type of bombardment employed in cyclotrons. Most of the radio isotopes obtained by bombardment are β–active, but some change into another element because the nucleus absorbs an outer electron from the K–level (K–capture). Isotope production by transmutation reactions is considered the special sphere of the cyclotron. Some of the results obtained by the authors in the Amsterdam synchrocyclotron are listed in a table partly reproduced below.

Yield of Radio Isotopes from Deuteron Bombardment at 30 MeV

Isotope	*Half-life*	*Radiation*	*Target material*	*Reaction*	$\frac{\mu c}{\mu Ah}$
^{7}Be	53 d	K, γ†	$LiBO_2$*	d, 2n	150
^{48}V	16 d	K, γ	Ti	d, 2n	350
^{51}Cr	26 d	K, γ	V	d, 2n	280
^{52}Mn	6 d	K, $\beta+$, γ	Cr	d, 2n	400
^{55}Fe	3 y.	K	Mn-Cu	d, 2n	10
^{58}Co	72 d	K, $\beta+$, γ	Zn	d, 2α	0.04
^{56}Zn	250 d	K, $\beta+$, γ	Cu	d, 2n	20
^{66}Ga	9.4 h	K, $\beta+$, γ	Zn	d, 2n	3,600
^{86}Rb	19 d	$\beta-$, γ	Sr	d, α	20
^{114}In	50 d	$\beta-$, γ	Cd	d, 2n	16

† K denotes the capture of a K-shell electron.
* Non-Metallic.

The time of bombardment in each case was 2 minutes. A special difficulty in target preparation is that of the heat evolved under irradiation. A beam current of 30 μA results in 900 W being converted continuously at the target. Due to the very high vacuum there is no cooling by convection. In some cases this makes it necessary to reduce the beam intensity in order to protect the target. In other cases special means of cooling can be applied with success. For instance, the metal plate forming the target is silver-soldered to a water-cooled copper pipe. This is satisfactory in the case of gold, silver and platinum. Where the material is too brittle to permit this technique it is silver-soldered to a copper plate which in turn is soldered to the copper tube, or the material is deposited on the copper plate in the form of a layer or alloyed with it. Radioactive phosphorous has been obtained by bombarding a plate of iron phosphide. Targets of some of the alkali metals, halogens, *etc.* have been prepared in the form of a glass applied as a thin layer about 0.5 mm. thick to the water-cooled copper tube. Where none of these preparation techniques is successful a perforated plate has been used which is joined to the water-cooled copper tube and which carries a series of small holes which are filled with the target material in powdered form. The separation of the radio isotopes from the targets is accomplished by various standard methods involving crystallisation, iron exchange, extraction and electrolytical methods, evaporation and distillation including vacuum distillation, co-precipitation, and paper chromotography and paper electrophoresis.

Sommaire: Description de la production de radio-isotopes avee le cyclotron d'Amsterdam, et détails sur la préparation des cibles et la séparation des radio-isotopes a l'etat pur.

Article by A. H. W. Aten & J. Halberstadt *Philips Tech. Rev.* **16**, July 1954 1-12

Importance of Thermal Equilibrium in Measurements on Powdered Insulators in Vacuo

188/III

See Abstract No.: 244/I

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Gas and Water Vapour Absorption in Liquids of High Boiling Point at Pressures below 10 mm. Hg

189/III

Germany. Vacuum procedures are employed on a steadily increasing scale in the chemical processing industry. A knowledge of the amount of gas contained in liquids, processed at reduced pressures, is of importance in connection with various types of production, *e.g.* in the electrical industry (oil impregnation of condensers and cables), or in distillation, or in vacuum drying. Methods of measuring the solubility of gases in liquids at atmospheric pressures are available and some details of one of these measuring methods are given. The authors report on a method developed for the determination of the solubility of gases in liquids subjected to reduced pressure varying from 0.01 to 10 mm. Hg. In order to be successful such a method must comply with the following requirements. (1) Its accuracy must be of the order of 10^{-5}mg/g. of the substance in order to determine any possible deviations from Henry's law. (2) The gas content of the sample has to be reduced to a value equivalent to an equilibrium pressure, not more than one order of magnitude below that of the gas phase at which the measurements are taken. (3) The method must ensure that the gas dissolved in the liquid is in equilibrium with the gaseous atmosphere outside the liquid. (4) The measuring plant must be free from any constructional materials which may produce gases by catalytical mechanisms. Details of an all-glass apparatus facilitating measurements under these specified conditions are given. Essentially the plant consisted of a distillation column where the evaporator was a glass spiral carrying the heater filament within, and situated in the centre of the column, the condenser being formed by the walls of the column which were water-cooled. The material to be degassed and/or tested was conveyed from a storage vessel to the bottom of the distillation column and the condensate was collected in a separate receptacle which had a connection with the feeding line to the distillation column; the latter could be sealed-off by a magnetically operated valve. The whole apparatus could be evacuated to 10^{-5}mm. Hg by a diffusion pump unit which contained in its backing line a calibrated collecting vessel which in turn was connected to a McLeod manometer which could be heated to prevent the condensation of water vapour in it. In operation the liquid was admitted to the apparatus and circulated through the column while the apparatus was pumped down. When a pressure of 10^{-3}mm. Hg had been obtained the temperature in the column was raised to the required level and the liquid was distilled over. Following this 'degassing' process the material was continuously circulated through the apparatus and the gas to be tested admitted through a needle valve at the required partial pressure. After saturation of the liquid with the gas the liquid was collected in the receptacle for the condensate, the receptacle was shut off by the magnetic valve and the remainder of the plant was re-evacuated to 10^{-3}mm. Hg. Subsequently the liquid was re-circulated through the system and degassed in the manner described previously. The gas evolved was collected in the calibrated vessel connected to the backing line of the pump and the amount collected was determined with the help of the McLeod manometer. The accuracy of this method is about $\pm 7.5\%$. The article concludes with a brief description of a large-scale degassing apparatus which facilitates degassing of liquids at a rate of 3 ton. per working day.

Sommaire: Description d'une méthode de mesure de petits quantités de gaz dissous dans des liquides traités sous basse pression.

Article by W. Oetjen & F. Gross *Chem. Ing. Tech.* **26**, Jan. 1954 9-13

Developments in Meat Dehydration in the United Kingdom

190/III

See Abstract No.: 171/III

Progress in Meat Dehydration

191/III

See Abstract No.: 75/IV

A Method for Protecting the Surfaces of Some Materials in Freeze Drying

192/III

See Abstract No.: 77/IV

An Efficient Cryostat for Producing Temperature between 4° and 80°K: The Production of Liquid Hydrogen Targets Using Liquid Helium

193/III

United States. Two cryostats are described for maintaining temperatures between the normal boiling point of helium (~4°K) and that of nitrogen (~80°K), one designed to maintain a high pressure vessel, with a heat intake of 10 cal./min. at 30°K, at any temperature between 4° and 80°K, the second to provide a liquid hydrogen target for the Harvard cyclotron at a constant temperature. Both cryostats are based on the same principle. Liquid helium is introduced to a reservoir whence it is pumped *via* a heat exchanger, in the form of a coil of copper tubing round the cryostatic vessel, and thence, *via* a needle valve, to a small vacuum pump. Evaporation occurs in the coil, extracting 20 cal./mole of helium and, during the warming up of the helium gas to the cryostatic temperature, a further 5 cal./mole per degree are extracted, so that, with a heat leak into the system, any desired temperature is available, merely by adjusting the flow rate. The high pressure cryostat consists of an all metal vacuum vessel, constructed so that liquid nitrogen shielding is provided by a re-entrant reservoir above, and extending around, the liquid helium pot which is supported by a filling tube passing up through the liquid nitrogen bath. A $\frac{1}{8}$–inch, copper-nickel, thin walled tube leads from the bottom of the liquid helium pot to the heat exchanger, consisting of a three layer coil of 3/16 copper tubing, silver soldered to a copper tubular core, which forms the wall of the cryostatic vessel. The heat exchanger tubing leads into the bottom closed end of this wall and the upper end is continued as a thin walled stainless steel tube through the upper wall of the high vacuum space, through the liquid nitrogen reservoir, to terminate in a room temperature seal

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to the emergent portion of the high pressure experimental chamber assembly. Just below this seal a side tube leads from the stainless steel tube to the needle valve and pump. A 1.5 l. filling of liquid helium is sufficient to cool the cryostat to 30°K and maintain it at that temperature for 3 hours. The liquid helium may be replenished during operation without closing down the system. Temperatures above 80°K, up to room temperatures, are available by using liquid nitrogen in place of liquid helium and not using the liquid nitrogen shield. The second cryostat, referred to as the helium–hydrogen convertor, was built to avoid the use of hydrogen liquefiers for safety reasons. The target consists of a 3 cm. diam. 17 cm. long tube of 0.0006 inch thick beryllium copper foil, joined to a cylindrical hydrogen liquefying chamber of about the same volume, totalling 300 c.c. Above the liquefying chamber is a long cylindrical liquid helium reservoir, of 4 l. capacity, suspended by its filling tube from a liquid nitrogen reservoir through which the tube leads out to room temperature, ending in a safety valve and filling enclosure. A fine, cupro-nickel tube leads from the bottom of the liquid helium reservoir to a refrigerating coil, consisting of about 4 m. of $\frac{1}{8}$–inch copper tubing, wound round the liquefying chamber, thence to a 2 m. coiled length of cupro-nickel tubing, soldered along its length to a similar tube carrying the incoming hydrogen gas to form a preliminary heat exchanger, and thence, together with the hydrogen line, through the liquid nitrogen reservoir to the exterior, where the hydrogen line is connected to its supply and the helium line, *via* a needle valve, to a mechanical vacuum pump. A tubular radiation shield at liquid nitrogen temperature encloses the liquid helium reservoir and target tube, having a large window of 0.0005 inch thick aluminium. A depth gauge, consisting of a stack of five 92Ω carbon resistors connected to a 6 V battery (the current increasing through a resistor, below which the liquid level has fallen, by almost 100%), is used to monitor the level of liquid helium. Thorough evacuation and flushing, prior to charging with hydrogen, is necessary to avoid clogging the tubes with solid nitrogen. The hydrogen system is precooled by pumping liquid nitrogen through the heat exchanger coils and 12 hours are required to cool the radiation shields. A few litres of liquid helium are then transferred and the needle valve opened to pump it through the coils till the liquefaction chamber is cooled sufficiently, more hydrogen is admitted and liquefied at an over-pressure of almost 1 atm., the 200 c.c. required taking about 2 hours. The loss of liquid helium to maintain the target is 150 c.c./hr. but this can be reduced by accelerating ortho-para conversion of the hydrogen and improving vacuum insulation.

Sommaire: Description de deux cryostats employés dans différents problemes, le premier maintenant des températures entre 4 et 80°K, le second la température d'une cible à environ 20°K.

Article by C. A. Swenson & R. H. Stahl
Rev. Sci. Instrum. **25**, June 1954 608-611

Steam Vacuum Refrigeration

194/III

United States. The principle of steam vacuum refrigeration, though known for over fifty years, was not extensively used until 20 years ago and has recently received renewed interest due to the expansion of the chemical and process industries and the need for refrigeration above 32°F, the lower limit of the method. The increased industrial use of vacuum techniques had a beneficial effect on steam ejector design as well and resulted in the development of installations of this kind which are appreciably more reliable and efficient today than in the recent past. Essentially, the method consists of lowering the temperature of water flowing through a tank by causing it to evaporate at, say, 45°F which is facilitated by reducing the pressure in the tank to 0.1475 p.s.i.a. with the help of a booster steam jet ejector fitted to the system. The ejector conveys the vapour to a main condenser which is in turn evacuated by a two-stage steam ejector with intercondenser to a pressure lower than that prevailing in the flask tank. The water in the flash tank, chilled by the evaporation process, falls to the bottom of the tank and is removed. The level of the water in the flash tank is maintained by suitable means. The lower the temperature to which it is desired to cool the water, the higher is the number of pounds of steam required per ton of refrigeration. Numerical values for this relationship are given in graphs. In a similar manner it is shown that the steam consumption drops as the condenser water temperature decreases at a given flow rate of condensing water. The steam consumption of both ejectors is reduced if higher pressures are used. Economies result from using ejectors designed for the highest possible pressures. Applications of the method to direct cooling of various fluids is considered as is also the cooling of corrosive gases and other steam jet refrigeration installations.

Sommaire: Revue sur le principe de fonctionnement et l'emploi des méthodes de refrigération par le vide a l'aide d'éjecteurs à vapeur.

Article by E. Spencer
Heating, Piping Air Condtng. August 1954 104-107

Improvements i.o.r.t. Means for Applying Pressure and Suction to Concrete Which is Being Moulded

195/III

New Zealand. The strength of concrete depends on its water-cement ratio. Initially the water content is kept high for the purpose of manipulating the mix and in order to facilitate moulding to the correct shape, but the ultimate strength of the moulded article depends on the density of the concrete, *i.e.* on close spacing of its constituent particles in the mass. Thus it is important to treat the concrete mass during the consolidation phase in such a manner that the diameter of the capillaries, through which excess water is withdrawn, is reduced to a minimum. Results superior to those obtained in the past are claimed to be achieved by a method whereby pressure and suction combined with some form of a filter are employed to remove the water in the consolidation phase. Prior to filling the mould with the concrete mix, a stout-walled rubber tube is placed in the centre of the mould, which is stiffened internally to prevent its collapse below a certain diameter, and is closed at one end. Longitudinal wires at the outer surface of the tube provide a path between the outer surface of the tube and a filter made from fabric which separates the tube and the wires from the concrete. The filter sleeve is pleated to allow for radial expansion of the rubber tube. After assembly of the components just described the mix is poured into the mould and a fluid under pressure is supplied to the bore of the rubber tube. As a result the latter expands radially and squeezes the surrounding concrete mass. The water squeezed out of the concrete collects in the space between the tube and the filter. In order to remove this water speedily, a vacuum pump is

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connected to that space. The suction produced by the pump, besides facilitating the speedy removal of the water, assists in the consolidation of the concrete. When the concrete has become sufficiently set to be self-sustaining, the pressure in the rubber tube is reduced to normal and the assembly can be withdrawn from the moulded article. The hole left in the moulded article will accommodate pre-stressed rods for post-tensioning by the conventional methods or can be employed for curing with hot gases or liquids.

Sommaire: Un béton particulierèment solide et densité élevée peut être obtenu en appliquant une pression et une succion par des moyens speciaux pendant la phase de durcissement.

L. R. Clapp
Brit. Pat. N.B. 713,969

Removing Carbon Bisulphide from Viscose Fibre with a Liquid Seal Vacuum Pump

196/III

United States. Wet acid regenerated cellulose yarn made by the viscose process has to be washed in hot water to remove the carbon bisulphide content and the mineral acid salts and other by-product impurities present. Cold-water treatment would be sufficient for the removal of acids and salts but for the presence of carbon bisulphide which acts as a barrier. A vacuum process is described whereby the original carbon bisulphide content of 16–18% is reduced to 4% or less thus permitting cold-water treatment of the material for the removal of the remaining impurities. The rayon filament tow to be treated is fed into a can with a false bottom about 2 inch. above the true bottom. The can holds a charge of about 3,000 lb. of wet acid tow in the form of a layer of about 33 inch. depth which is equivalent to 600 lb. of production. Due to the presence of the false bottom evaporation from the layer can take place from the top surface and from the bottom surface. The can is placed into a vacuum chamber which is connected to a water ring pump. The pump is run in conditions where the sealing water of the pump is kept at about 50°C, *i.e.* the pressure obtained in the chamber is comparatively high, *i.e.* about 5.5 inch. Hg. These operating conditions were chosen because it had been found that running the pump at the usual high vacuum of about 2 inch. Hg resulted in excessive evaporation of water from the processed material which in turn produced appreciable cooling of the processed material and an unfavourable proportion of water vapour and carbon bisulphide vapour was extracted from the material. At the same time the operational temperature of the sealing water was high enough to prevent condensation of the carbon bisulphide in the pump, eliminating the danger of corrosion. Vapours leaving the pump are passed into a separator. From here the carbon bisulphide vapours are conveyed to a separate recovery system and the water returned to a storage tank from which, by means of a centrifugal pump, it is passed through a heat exchanger where its temperature is re-adjusted to 50°C and from which it is returned to the liquid ring pump. Data of a typical run is given as follows.

Staple Tow Temp. °C:	
Start	37.5
End	32.5
Evacuation Time, minutes	90
Minimum Absolute Pressure, inches Hg	5.5
Weight Sample (in pounds-dry basis)	277
Per cent. CS_2 in sample after evacuation	1.4
Treatment (based on dry sample) Seal Water, temp. °C	50
Per cent. CS_2 in Wash Effluent:	
end 1 hr. wash	.037
end 2 hr. wash	.022
end 2½ hr. wash	.015

Following this process the material is treated for about 5 hours by washing in water at 35–40°C which satisfactorily removes the last traces of carbon bisulphide and acids and salts.

M. C. Hall & E. I. Du Pont de Nemours & Co.
U.S. Pat. 2,673,785

Sommaire: Description d'une méthode à vide pour la récupération de bisulphate de carbone à partir de fils de cellulose humides, obtenus par le procédé "viscose".

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40 — GASES AND VAPOURS — 40

Gasometric Method for Determination of Hydrogen in Carbon 63/IV

See Abstract No.: 155/II

Manipulation of Radioactive Gases in High Vacuum Apparatus 64/IV

See Abstract No.: 186/III

Determination of Tritium by Ion Current Measurement 65/IV

United States. Tritium serves as a useful tracer in biological and chemical studies, but its use is limited by the difficulties experienced in its determination on account of the low energy of the β–particle emitted during disintegration (approximately 18.9 keV). For instance, end-window counters and windowless counters are ineffective for this purpose. The method described in this article facilitates the determination of 3×10^{-10} to 0.01 curie of tritium in gases, which are readily prepared from water or organic compounds, with an accuracy of 1%. It provides for the gas to be introduced into the detector, in this case a Borkowski ionisation chamber, and measuring the ion current by means of a vibrating reed electrometer—a method which has proved to be satisfactory in carbon –14 analysis. The ionisation chamber employed had a volume of 250 c.c., was 7 cm. long and was made from stainless steel. The chamber was mounted on the electrometer and the output of the electrometer was recorded on a strip-chart potentiometer. After evacuating the chamber to 10^{-3} mm. Hg or less, the sample of the gas to be assayed was expanded into the chamber or transferred into it by a Toepler pump, giving a pressure of approximately 1 atm. The chamber walls were charged with a voltage negative with respect to earth and low enough to avoid multiplication of ions. An ion current less than 10^{-13} A is measured by the rate of charge of a capacitance of about 12pF within the electrometer; a current greater than 10^{-13} A is measured by the voltage drop across a resistor. Measurements of relative ion current can be converted to values of absolute ion current by calibration with a sample containing a known amount of tritium. The filling gas, pressure and the collecting voltage have an influence on the magnitude of the ion current collected. The ion currents collected in several gases from small amounts of hydrogen-tritium mixture in chambers filled to atmospheric pressure were measured at saturation voltage. The results relative to those in methane are given in a table reproduced below. Comparison of these values with the respective data given by Valentine and

Ionisation Collected from Tritium Radiation in several Gases

Gas	*Rel. Ionisation*	*Ion Pairs/ Disintegration*
Hydrogen	0.685	132
Nitrogen	0.82	158
Helium	0.84	162
Carbon Dioxide... ...	0.84	162
Methane	1.00	193
Argon	1.06	205
Propane	1.13	218

Curran indicate a chamber efficiency of more than 95%. The factor facilitating the conversion of ion current measurements to disintegration rate was determined for methane, using a sample of known disintegration rate in two experiments, as 3.04×10^{-17} and 3.14×10^{-17} coulomb/disintegration at a half life of 12.4 years. Employing the average of these two values and the data shown in the previous table the relationships shown in the following table have been established.

Relation of Ion Current to Disintegration Rate

Gas	*Coulombs/Disintegration* $\times 10^{17}$	*Curies/Ampere* $\times 10^{-6}$
Hydrogen ...	2.12	1.275
Methane	3.09	0.874
Propane	3.49	0.775

Article by K. E. Wilzbach, A. R. van Dyken & L. Kaplan *Analyt. Chem.* **26**, May 1954 880-883

Sommaire: Description d'une méthode normalement employée pour le carbone –14 qui permet de déterminer la présence de tritium dans des gaz pouvant être obtenus à partir de composés organiques.

Diffusion Coefficient, Solubility, and Permeability for Helium in Glass 66/IV

See Abstract No.: 157/II

Adsorption of Helium on Glass at Temperatures Below the λ–Temperature 67/IV

Holland. The adsorption of helium on glass was measured at temperatures below 2.19°K. A novel differential-pressure gauge, accurate to 0.1 microns at a total pressure of a few mm. of Hg, is described. No temperature

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dependence was detected for the isotherms in the range 1.54°K to 2.19°K. The number of adsorbed statistical layers n is given as a function of the reduced vapour pressure $p/p_{sat.}$ n remains comparatively small even for $p/p_{sat.}$ as high as 0.99, in general agreement with the results of Brewer and Mendelssohn, and Bowers. The results are not in accord with those of Kistemaker, and Long and Meyer.

(*Chemical Abstracts*)

Sommaire: On a mesuré l'adsorption d'hélium sur du verre à des températures au dessous de 2.19°K. On décrit une nouvelle jauge differentielle précise au 0.1 microns pour une pression totale de quelques mm. Hg.

Article by H. H. Tjerkstra, F. J. Hooftman & C. J. N. van den Meydenberg *Physica* 19, 1953 935-942

A New Type of Helium Liquefier 68/IV

See Abstract No.: 169/II

The Organisation and Purpose of the Liquid Helium Pool 69/IV

United Kingdom. The Liquid Helium Pool is a service for supplying liquid helium by road or rail in special Dewar vessels to any laboratory equipped to make use of it. This may have a great effect on the future of low temperature research in this country. The purpose of this paper is to describe the organisation and finances of the scheme, together with some of the techniques and equipment used.

(*Author*)

Sommaire: Le 'Liquid Helium Pool' est une organisation pour mettre en commun la liquéfaction de l'hélium. On décrit l'organisation et son financement, ainsi que les techniques et les installations necessaires.

Article by E. Mendoza *Vacuum* 4, Oct. 1954 476-485

41 — FREEZE DRYING — 41

Preservation of Bacteria 70/IV

See Abstract No.: 255/I

A New Approach to Freeze Drying of Tissue 71/IV

United States. With the conventional techniques of freeze drying tissue, 4–24 hours are required to desiccate specimens of animal tissue. Plant tissue requires a longer period. The apparatus described in this paper reduces the drying time of plant tissue to 2–8 hours. This is effected by allowing a stream of dry air at –30°C or lower to pass over the samples during drying at a pressure of 1.5 cm. Hg. The air is drawn through a flowmeter, over a chemical desiccant and through a cooling device of special design into the drying chamber. The cooling device is of a shape similar to a re-entrant trap, immersed in a dry ice–methyl cellosolve mixture at a temperature of –75°C. In order to ensure proper drying of the air en route to the drying chamber, the air passes through a coil surrounding the cylindrical cooling device before it is conveyed to the drying chamber. Here for the same reason the air is passed first through a coil surrounding the cylindrical drying chamber immersed in a cool temperature bath before it is admitted to the bottom of the latter. The drying chamber is kept at a temperature of –30 to –40°C using the same refrigerant as in the case of the air-cooling device. Inside the drying chamber and close to the bottom, a fritted glass plate is inserted to ensure proper distribution of the incoming air and to serve at the same time as a support for the plastic holder carrying the tissue specimens inserted in small metal containers. The upper end of the drying cylinder is closed with a brass plate by means of an 0–ring seal, and connection to the pump is made *via* an outlet near the top of the drying chamber. A water trap is inserted in the pumping line. A stopcock in the air line facilitates control of the air flow. The drying chamber has a diameter of about 3 inch. and a length of about 14 inch. The whole apparatus is made from glass. Stem tissue dried in the apparatus lost up to 90% of its water content in the first two hours and was oven-dry after about four hours. The arrangement described eliminates the need for a diffusion pump. As the drying process is isothermal there is no need for a heater.

Sommaire: Description d'une nouvelle méthode de lyophilisation de tissus sous pression relativement élevée donnant un courant d'air sec continu qui réduit ainsi le temps de séchage.

Paper by W. A. Jensen *Exp. Cell Res.* 7, 1954 572-574

The Mechanism of Drying of Solids: Part V: Freeze Drying of Clay and Vacuum Drying of Wet Clay 72/IV

See Abstract No.: 218/I

The Effects on Biological Materials of Freezing and Drying by Vacuum Sublimation. I. Development and Testing of Apparatus 73/IV

United States. The freeze drying technique has been used for some time for the preservation of biological materials such as bacteria and virus, but only recently have investigations been started into the effects of design and performance features of freeze drying equipment on the survival rate of the micro-organisms processed. Many of the methods used in the past show limited versatility, falling short of the requirements for an accurate study of the effects of low temperature desiccation on survival of viruses and cells. These requirements are listed by the authors as follows: Removal of water molecules at temperatures ranging down to

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–80°C, continuous operation with the minimum of attention by the operator, sealing-off of samples at operating pressure, processing of different samples of the same material at different temperatures simultaneously, isolation of a portion of the apparatus without affecting the remainder of the system, and finally, determination of the end-point of sublimation without removing samples from the system. The authors describe an arrangement which was designed to meet these conditions. The system is made of glass throughout and uses high vacuum stopcocks and Apiezon grease at all joints. It can be exhausted to pressures below 10^{-5} mm. Hg facilitating sublimation at –80°C, at which temperature the vapour pressure of ice is 4×10^{-4} mm. Hg. Evacuation is effected by an oil diffusion pump, Narcoil 20 being employed as the working fluid which proved less tarry and gummy when exposed to atmosphere at operational temperatures than Octoil gas. Mercury diffusion pumps proved unsuitable due to backstreaming with the attendant deleterious effect of mercury on the enzyme systems of the processed material. The apparatus consisted of a primary and a secondary manifold. The former, to which the pump was connected, was fitted with a cold trap, immersed in a Dewar flask containing dry ice and acetone (–80°C), and an ionisation gauge. The second, joined to the first *via* a stopcock, consisted essentially of a large liquid-air operated moisture trap serving as the condenser and means for connecting the lyophilisation tubes. The latter consisted of Pyrex test tubes with long narrow necks and male standard taper joints. Each pair of tubes assembled to the system could be immersed in a single Dewar flask and thereby kept at any desired temperature independently of the rest of the tubes being processed simultaneously. The temperature at the condenser was –190°C, and under normal operational conditions the liquid air charge had to be replaced every 24 hours. On completion of the drying, the heavy wall of the narrow necks of the tubes facilitated sealing off by flame at the operational pressure in the second manifold, *i.e.* 10^{-5} mm. Hg as long as the bottom of the tube was cooled. The dehydration of 0.3 ml. of virus suspension at –80°C in the plant described took about 8 days. By introducing a modification, details of which are given, this time could be shortened to 2 days. In conclusion the authors report briefly on experiments, in the course of which the ionisation gauges attached to the primary and secondary manifolds have been employed successfully for the determination of the end-point of the sublimation.

Sommaire: On donne des détails sur un appareil expérimental de lyophilisation, spécialement éxécuté pour faciliter les études méthodiques des effets des différents facteurs de cette méthode, sur le taux de survie des micro-organismes traités.

Article by D. Greiff & H. Pinkerton *J. Exp. Med.* **100**, July 1954 81-88

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The Effects on Biological Materials of Freezing and Drying by Vacuum Sublimation. II. Effect on Influenza Virus

74/IV

United States. Abstract **73/IV** gives a description of an apparatus designed and constructed to facilitate methodical studies of the various factors of the freeze drying method on the survival rate of virus suspensions dehydrated by this technique. The present article reports on the results obtained in such a study using influenza virus suspensions as the processed material, *i.e.* PR8 strain of influenza A virus kept in fertile eggs for several years. (*a*) *Effects of repeated freeze-thaw cycles.* Allantoic fluid from the infected eggs was deep frozen at –40°C, withdrawn after solidification and allowed to melt at room temperature. Repeating this cycle five times resulted in the infectivity titre falling from $10^{-8.6}$ to $10^{-0.8}$, cycles 2, 3 and 4 causing greater losses than cycles 1 and 5. It is suggested that seed crystals are present in this suspension after the first cycle resulting in the formation of numerous small crystals in the second and subsequent cycles, capable of injuring the virus particles mechanically. In that case the reduction of the loss in the fifth cycle could be explained by a reduced number of live virus particles present. (*b*) *Effects of speed of freezing and storage temperature.* Details of the experimental procedure are given. Rapid cooling to –40°C or slow cooling to –80° or –190°C did not cause any significant loss but rapid cooling to above –40°C or slow cooling to above –80°C definitely caused a loss. Storage at temperatures above –40°C caused a loss of titre irrespective of the temperature to which the material was cooled originally. Examination with polarised light showed the specimens, which were cooled rapidly to –80 or –190°C, to be vitrified. Other modes of cooling resulted in the formation of ice crystals to a varying extent. (*c*) *Effects of preliminary treatments and sublimation temperatures.* The effects on the infectivity titre are shown in a table partly reproduced below.

The Effects of the Preliminary Treatment and of the Sublimation Temperature on the Preservation of Influenza Virus

Preliminary treatment	*Sublimation temperatures*					
	20°C	0°C	—10°C	—30°C	—60°C	—80°C
Snap frozen† from 20°C ...	—4.0*	—2.5*	—3.2*	—3.7*	—2.4*	—2.7*
Snap frozen from 0°C ...	—4.0	—2.3	—3.0	—3.5	—2.3	—2.0
Slow frozen at —30°C ...	—3.0	—2.0	—	—2.7	—2.5	—2.6
Slow frozen at —80°C ...	—2.0	—0.5	—	—2.4	—2.0	—0.6
Slow frozen at —190°C ...	—1.5	+0.8	—	—1.4	—1.0	0.0
Fast frozen at —30°C ...	—3.0	—1.4	—	—2.8	—2.5	—2.4
Fast frozen at —80°C ...	—1.5	+0.6	—	—1.7	—1 0	+0.2
Fast frozen at —190°C ...	—1.2	+0.6	—	—1.4	—0.7	+0.8

* Figures indicate loss (—) or gain (+) in infectivity titre (expressed in logs) at the end-point of vacuum sublimation as compared with non-sublimated controls.

† The term 'snap freezing' refers to freezing of the virus suspension within the vacuum system by a rapid lowering of the pressure.

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The infectivity titres of the controls and tested samples were similar after dehydration at 0°C following slow cooling to –80°C or in the case of dehydration at –80°C following rapid cooling to –80°C or slow cooling to –190°C. All other combinations of speed of freezing and sublimation temperatures resulted in a marked loss. The most unfavourable method of freezing was 'snap freezing'. The least effective sublimation temperature was + 20°C. In general, drying from the vitreous state gave better results than drying from the crystalline state.

Sommaire: L'appareil décrit dans l'article 73/**IV** a été employé pour étudier les effets des différents facteurs de ce procédé sur le taux de survie du virus de la grippe.

Article by D. Greiff, H. Blumenthal, M. Chiga & H. Pinkerton *J. Exp. Med.* 100, July 1954 89-101

Progress in Meat Dehydration

75/IV

United States. Freeze drying, which has been used for fifty years in the biological field, is now being experimentally applied to the preservation of meat. Drying from the frozen state under vacuum allows little loss of volatile components and gives a porous structure which is easily rehydrated. The freezing before drying inhibits chemical, enzymatic and bacterial change, and the low residual moisture content after drying prevents these reactions from resuming when the temperature is raised. Experimental work with bulk and patty hamburger, steaks and chops has been undertaken in three types of commercially available dryers: A plasma dryer using infra-red heating and operating at 0.2 mm. Hg pressure, a vacuum oven with water-heated shelves and using steam ejectors for the water vapour removal operating at 0.5 mm. Hg and a third, which operates at 0.3 mm. Hg pressure, having the same heating arrangement and a refrigerated surface in the base of the chamber serving as a vapour trap. The meat is pre-frozen before drying either in a dry-ice chest or by means of mechanical refrigeration at –10 to –30°F. None of the differences obtained in the finished product could be traced to variations in handling before drying. Drying times varied from 8 hours in the plasma dryer to 72 hours in the steam ejector oven, the latter time being greatly in excess of that actually required. The table reproduced in part below shows the final moisture content of various products dried for several time-temperature combinations.

Relation of Time, Temperature, and Vacuum to Final Moisture Content of Various Meats

Product	*Drying Time (Hr.)*	*Maximum Oven Temp. (°F Time)*	*Vacuum (Microns)*	*Moisture (%)*
Steak	27	105°/12 hr.	500-700	2.6
Hamburger Patties	72	100°/48 hr.	300	1.9
Pork Chops ...	72	100°/48 hr.	300	2.5
Hamburger ...	60	95°/50 hr.	300	1.2
Steak	60	95°/50 hr.	300	1.5
Steak	48	120°/40 hr.	300	1.0

A study of the moisture absorption of the dried material indicated that a moisture-proof wrapper is required. The quality also deteriorates if the product is stored in air. Packing the dried meat in flexible material was unsuccessful partly because of bone puncturing. Storage in evacuated cans is preferable. Re-hydration by immersing in tap water at between 70 and 100°F for 20–30 minutes returned the meat to its original texture but not to its original colour. Some of the fresh meat odour returned on re-hydration and the odour was normal during drying. The palatability of the samples varied considerably, but in general, hamburger dehydrated in bulk fared best and possessed acceptable flavour after two years storage. A whey-like odour became more pronounced with increase of storage time of the dried meat, but there was insufficient evidence to connect this odour with 'off-flavour' development.

Sommaire: On fait un rapport sur l'état actuel des recherches sur la lyophilisation de viande de différentes espêces.

Article by H. P. Furgal *Food Engng.* 26, Sept. 1954 74-76, 152

Caution in the Use of Liquid Propane for Freezing Biological Specimens

76/IV

See Abstract No.: 158/II

A Method for Protecting the Surfaces of Some Materials in Freeze Drying

77/IV

Australia. The freeze drying of apricot and peach purées, whereby the purées were frozen before loading into the dryer, yielded a product which was not uniform in texture. This was traced back to the formation of gummy films of the processed material, which is most likely due to the large quantities of sugar contained in the processed material. These difficulties have been overcome by forming layers of pure ice above and below the material in the freezing stage prior to drying.

Sommaire: Un manque d'homogénéite de la texture près des surfaces et une adhésion solide sur les plateaux de quelques matériaux, apparaissent à la lyophilisation des purées de fruit. Ces difficultés ont été évitées en formant des couches fines de glace pure au dessus et au dessous du produit à lyophiliser.

Letter by J. D. Mellor *Vacuum* 4, July 1954 341

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45 — ELECTRONICS — 45

78/IV

Ion Source

United States. An ion source is described to be used for the copious production of ions in an isotope separating device which is suitable for producing isotopes on a commercial scale of materials such as copper –63 and –65, cobalt –59 and –60 or uranium –234, –235, –237, –238. The source of electrons consists of a massive thimble-like main cathode, inside which is supported an auxiliary-cathode, both made from tungsten. Around the outside of the main cathode is placed a filament which is heated to a temperature sufficiently high to initiate electron emission from the main cathode. A source of alternating voltage is connected between the main and auxiliary cathode. When electron emission from the main cathode has been established the alternating voltage ensures that the arc discharge between the main and auxiliary cathode is maintained, and the filament is then switched off. The external supply circuit is arranged so that the power input to each of the electrodes can be controlled independently of the other electrode. The anode consists of a platform of tungsten rods placed near to the cathode structure such that the auxiliary cathode is shielded from the main discharge by the massive effect of the main cathode. Metal is fed to the arc through a tube, the end of which is directed towards the anode platform. The metal is introduced in the form of a wire or ribbon and may be continuously or intermittently fed. Ions produced in the main discharge are directed into the ion or isotope separating device by application of a large negative voltage to a cylindrical electrode placed coaxially with the arc cathode, between the latter and the isotope separating device. The whole source is mounted in a vacuum chamber.

C. M. Turner & U.S. Atomic Energy Comm. *U.S. Pat.* 2,679,597

Sommaire: On donne des détails sur une source d'ion employée dans des appareils séparateurs d'isotope, executés pour produire des isotopes sur une échelle commerciale.

79/IV

Method and Apparatus for Producing Ions

United States. Isotope separators, in which a beam of ions formed from the element whose isotopes are to be resolved is bunched in accordance with the mass of the ions present and then deflected in r.f. fields to direct the preferred enriched groups of ions into a collector, require ion beams of very high content before a substantial yield is obtained. Also, the ion source must be capable of continuous operation for a considerable period. A vacuum arc source, which fulfils both these requirements, is described. A hollow cylindrical anode mounted, with its axis vertical, above a flat indirectly heated anode having a central hole, is, in turn, set below a device for dropping a steady stream of powdered material axially through the assembly. This powder is vaporised by the radiant heat from the anode and the gas is ionised in the arc Any powder remaining falls through the hole in the cathode, below which is an offset, directly heated, filamentary cathode which is used to bombard the flat cathode and raise it to electron emissive temperatures. A magnetic device is used to vibrate the powder in the powder magazine, which forms part of the vacuum system, to prevent clogging of the material and interruption of the flow. In order to produce electrons at a high positive potential relative to the accelerating electrode of the analysing system, a cylindrical sleeve of insulating material is placed between the ion source and the tube containing the accelerating electrode. Both the tube and the electrode can then be earthed to provide the field-free region necessary for the flow of the ions withdrawn and accelerated by the electrode. The supports for the anode and cathode are water-cooled and a radiation shield is placed between the anode and the flange carrying the powder source, to prevent overheating.

J. E. Mack & U.S. Atomic Energy Comm. *U.S. Pat.* 2,690,515

Sommaire: Description d'une source d'ion employée dans des séparateurs d'isotope dans lequel la source de métal d'ou l'isotope choisi doit être séparé, est introduite dans l'arc sous forme de poudre.

80/IV

Proposal of Synchrotron with a Double Vacuum Chamber

Italy. A synchrotron with a double vacuum chamber is proposed which could compete with the strong-focusing synchrotron in the 10–beV region. The particles are injected into the large vacuum chamber and accelerated to a moderate energy. As soon as the particles reach a certain energy the beam will slide into the smaller chamber where the particles will be accelerated up to the maximum flux density. Several problems yet to be solved are briefly discussed. (*Nuclear Science Abstracts*)

Article by G. Salvini *Nuovo cimento* 11, May 1954 555-558

Sommaire: On propose un synchrotron avec une double chambre à vide qui pourrait entrer en compétition avec le synchrotron à forte focalisation dans le region de 10–beV.

81/IV

Performance Characteristics of Barium Getters with Particular Reference to Their Application in Thermionic Valves

Italy. The performance characteristics of pure barium getter deposits (bright mirrors only) evaporated from a barium-aluminium alloy have been investigated. In the introduction an outline is given of the task of the getter in all phases of the production and working life of a thermionic valve. The capillary method was used in the experiments but whereas previous workers conducted this test with the pressure in the manifold being constant, the author modified the equipment to facilitate accurate operation of the test with the pressure in the getter chamber constant. The results thus obtained are independent of specific design features of the test employed. It is proposed to assess getter performance in terms of 'instantaneous absorption capacity' expressed in litre

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micron per second. This facilitates in addition the calculation of the total absorption capacity of the getter by integrating the curve giving the instantaneous absorption capacity values obtained by measurement. If presented in this manner the results of such investigations are of immediate assistance for industrial practices. The behaviour of the getters towards a number of pure gases has been re-examined employing the revised capillary method. (See Abstract 81a/IV).

Sommaire: Les caractéristiques de performance des dépôts getter de baryum pur, (dépôts brillants seulement), évaporés à partir d'un alliage d'aluminium et de baryum à la température ambiente, ont été étudiées.

Article by P. della Porta *Vacuum* 4, July 1954 284-302

Performance Characteristics of Barium Getters at Elevated Working Temperatures of the Valves 81a/IV

Italy. The performance characteristics of pure barium getter deposits (bright mirrors only) evaporated from barium aluminium alloys in the temperature range of 100–400°C have been investigated and the results are compared with measurements taken at 20°C. (See Abstract 81/IV.) A rise in the operational temperature is shown to have a considerable effect on gas absorption. In the higher temperature regions the getter absorbs gas in quantities closely approaching those required for the formation of the respective chemical compounds. Elevated temperatures appear to cause the molecules of gases, forming an impermeable layer on the getter and thus poisoning it at room temperature, to diffuse into the body of the barium deposit removing such barrier layers entirely. Regeneration of exhausted mirror deposits by means of a heat treatment has been studied. The behaviour of barium mirrors evaporated on glass surfaces heated at 200°C has been examined and some comparative studies have been made between the absorption capacity of freshly deposited mirrors after heat treatment and prior to such treatment. The article concludes with observations on gas evolution from getters.

Sommaire: Les caractéristiques de performance des dépôts getter de baryum pur, (dépôts brillants seulement), évaporés à partir d'un alliage d'aluminium et de baryum a des températures de 100 a 400°C ont été étudiées.

Article by P. della Porta *Vacuum* 4, Oct. 1954 464-475

Application of a Tracer to Cathode-Gettering and Gas-Adsorption Problems 82/IV

See Abstract No.: 212/I

47 — GLASS PROCESSING — 47

Method for Sealing-off an Evacuated Component Fitted with a Thick-Walled Pumping Stem 83/IV

Holland. Evacuated components such as radio receiver valves fitted with thin-walled pumping stems present no difficulties with regard to the sealing-off of the latter. In the case of T.V. tubes however the pumping stems are thick-walled and the normally employed manual method of sealing-off by means of a flame usually results in releasing a certain amount of gas in the final stages of sealing to the detriment of the residual atmosphere in the tube. Also there is a high incidence of cracks occurring during this operation. An improved method of sealing-off the pumping stem is described which makes the whole operation safer. A tubular insulating component carrying a resistance winding on the inside is slipped over the stem and placed in a position covering the area where the stem is to be sealed off. The resistance winding can be connected to a supply in such a manner that either one half or the whole of the winding is heated. In order to effect closure of the stem in the desired place one half on the winding is connected to the supply warming up the section to be sealed to 380°C for a period of six minutes. Then the load is increased from 40 W to 150 W for a period of about 25 sec. which results in the immediate closure of the pumping stem. At this juncture the whole of the winding is incorporated in the electrical circuit and the load reduced to 28 W. As a result, the area adjacent to the point of closure of the stem is warmed up and this facilitates uniform and stress-free cooling. After a period of six minutes the heater element is removed and the surplus length of the stem can be cut off by conventional means.

Sommaire: On donne des détails sur une nouvelle méthode par laquelle les queusots à paroi épaisse des tubes de T.V. peuvent être scéllés sans aucun danger de félure.

W. A. Rooyers N. V. Philips Gloeilampenfabrieken *German Pat.* 889,826

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A.B.M.	Associaco Brasiliera de Metais, Via Duto, Donna Paulina 80-8°, Andar (Palocio Maua), Sao Paulo, Brazil.
Acta Metallurg.	Acta Metallurgica, 57, East 55th Street, New York 22, N.Y., U.S.A.
Acta Phys. Austriaca	Acta Physica Austriaca, Springer Verlag, Mölkebastei 5, Vienna, 1, Austria.
A.E.C. Rep.	U.S. Atomic Energy Commission Reports, Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
A.E.R.E. Rep.	U.K. Atomic Energy Research Est., Harwell, Berks. England, Her Majesty's Stationery Office, P.O. Box 569, London, S.E.1., England.
Amer. J. Med Sci.	American Journal of Medical Science, Lea & Febiger, Philadelphia, 6, Penn, U.S.A.
Amer. J. Phys.	American Journal of Physics, American Institute of Physics, Michigan State College, East Lansing, Michigan, U.S.A.
Analyst	The Analyst, The Society of Public Analysts, 7-8, Idol Lane, London, E.C.3, England.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Ann. Phys. Paris	Annales de Physique, Librarie Maison et Cie, 120, Boulevard St. Germain, Paris 6, France.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
Ark. Zoologi	Arkiv for Zoologi, Almquist & Wiksells Boktryckeri AB, Stockholm, Sweden.
Autom. Industr.	Automotive Industry, Chiltern Press, Chestnut and 56th Street, Philadelphia, 39, U.S.A.
A.W.R.E. Rep.	U.K. Atomic Weapons Research Estab. Aldermaston, Berks., Her Majesty's Stationery Office, P.O. Box 569, London, S.E.1, England.
Ber. Deutsche Ker. Ges.	Berichte Deutsche Keramische Gesellschaft, e.V., Poppolsdorfer Allee 15, Bonn, Germany.
Brit. J. Appl. Phys.	British Journal of Applied Physics, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Bull. Classe Sci.	Bulletin de la Classe des Sciences, Academie Royale de Belgique, Palais des Academies, 1, Rue Ducale, Bruxelles, Belgium.
Bull. Math. Biophys.	Bulletin of Mathematical Biophysics, University of Chicago Press, 5750, Ellis Avenue, Chicago 37, U.S.A.
Bull. Inst. Metals	Bulletin of the Institute of Metals, The Institute of Metals (Inc.) 4, Grosvenor Gardens, London, S.W.1, England.
Business Wk.	Business Week, McGraw-Hill Publishing Co. Inc., 99-129, North Broadway, Albany, N.Y., U.S.A.
Canad. J. Phys.	Canadian Journal of Physics, The National Research Council, Ottawa, Canada.
Canad. Mach. & Manufact. News	Canadian Machinery and Manufacturing News, Maclean-Hunter Publishing Co. Ltd., 418, University Avenue, Toronto, Ontario, Canada.
Ceramic Age	Ceramic Publishing Co. Inc., 421, Parker Street, Newark 4, N.J., U.S.A.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Chem. Engng. Progr.	Chemical Engineering Progress, American Institute of Chemical Engineers, 120, East 41st Street, New York 17, N.Y., U.S.A.
Chem. & Industr.	Chemistry and Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Verlag Chemie G.m.b.H., Weinheim/Bergstrasse, Haupstrasse 129, Germany.
Chem. Technik.	Chemische Technik, VEB Verlag Technik, Unter Den Linden 12, Berlin, N.W.7, Germany.
Compr. Air Mag.	Compressed Air Magazine, Compressed Air Magazine Co., Phillipsburg, N.Y., U.S.A.
C.R. Acad. Sci. Paris	Comptes Rendus des Séances de L'Academie des Sciences. Messrs. Gauthier-Villar, 55, Quai des Grands-Augustine, Paris, France.
Electronics	Electronics, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Elcctronic Engng.	Electronic Engineering, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.

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Elektrotech. Z.	Elektrotechnische Zeitschrift, Verband Deutscher Elektrotechniker (VDE) Verlag, Wuppertal, Germany.
Endeavour	Imperial Chemical Industries Ltd., 26, Dover Street, London, W.1, England.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Eng. News Rec.	Engineering News Record, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Exp. Cell Res.	Experimental Cell Research, Academic Press Inc., 125, East 23rd Street, New York 10, N.Y., U.S.A.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Food Technol.	Food Technology, Institute of Food Technologists, 119-123, West Park Avenue, Champaign, Ill., U.S.A.
Food Trade Rev.	Food Trade Review, Food Trade Review Ltd., 7, Garrick Street, London, W.C.2, England.
Glas-und Hochvakuum Tech.	Glas und Hochvakuum Technik. Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Heating, Piping Air Condtng.	Heating, Piping and Air Conditioning, Keeney Publishing Co., 6, North Michigan Avenue, Chicago, U.S.A.
Ind. Parfum.	Industrie de la Parfumerie, Press Documentaire, 28, Rue St. Dominique, Paris 7, France.
Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Inst. Chem. Engrs.	Institute of Chemical Engineers, 56, Victoria Street, London, S.W.1, England.
Inst. Refrig.	The Institute of Refrigeration, Dalmeny House, Monument Street, London, E.C.3, England.
Instruments	Instrument Publishing Co. Inc., 921, Ridge Avenue, Pittsburg 12, Pa., U.S.A.
Instrum. Mfg.	Instrument Publishing Co. Inc., 921, Ridge Avenue, Pittsburg, 12 Pa., U.S.A.
J. Amer. Oil Chem. Soc.	Journal of the American Oil Chemists Society, The American Oil Chemists' Society, 35, East Wacker Drive, Chicago 1, Ill., U.S.A.
J. Appl Phys.	Journal of Applied Physics, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Brit. I.R.E.	Journal of the British Institution of Radio Engineers, British Institution of Radio Engineers, 9, Bedford Square, London, W.C.1, England.
J. Chem. Phys.	Journal of Chemical Physics, American Institute of Physics, Inc., 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Chim. Phys.	Journal de Chimie Physique et de Physico-Chimie Biologique, Societe de Chimie Physique, 10, Rue Vauquelin, Paris (5e), France.
J. Exp. Med.	Journal of Experimental Medicine, Rockefeller Institute for Medical Research, York Avenue and 66th Street, New York, U.S.A.
J. Inst. Brewing	Journal of the Institute of Brewing, Brewing Industry Research Foundation, Nutfield, Surrey, England.
J. Inst. Metals	Journal of the Institute of Metals, The Institute of Metals (Inc.), 4, Grosvenor Gardens, London, S.W.1, England.
J. Inst. Petrol	Journal of the Institute of Petroleum, 26, Portland Place, London, W.1, England.
J. Lab. Clin. Med.	Journal of Laboratory and Clinical Medicine, 3207, Washington Blvd., St. Louis, Missouri, U.S.A.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers Inc., 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Phy. Soc. Japan	Journal of the Physical Society, Japan. Faculty of Science, Tokio, Japan.
J. Res. Nat. Bur. Stand.	Journal of Research of the National Bureau of Standards, U.S. Government Printing Office, Washington 25, D.C., U.S.A.
J. Sci. Instrum.	Journal of Scientific Instruments, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.

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J. Sci. Res. Inst. Japan	Journal of the Scientific Research Institute, Scientific Research Institute Ltd., 31, Kamifujimac-cho, Komagomc, Bunkyo-ku, Japan.
K. Fysiograf. Sällskapets Handlingar	Kungl. Fysiografiska Sallskapets Handlingar, Histological Institute, University, Lund, Sweden.
K. Fysiograf. Sällskapets I. Lund. Forh.	Kungl. Fysiografiska Sallskapets I Lund Forhandlingar, Histological Institute, University, Lund, Sweden.
Lab. Practice	Laboratory Practice, United Trade Press Limited, 9, Gough Square, Fleet Street, London, E.C.4, England.
Lancet	The Lancet Limited, 7, Adam Street, Adelphi, London, W.C.2, England.
Light Metals	Temple Press Ltd., Bowling Green Lane, London, E.C.1, England.
Machinery	Machinery Publishing Co., Clifton House, 83-117, Euston Road, London, N.W.1, England.
Mech. Engng.	Mechanical Engineering, American Society of Mechanical Engng. 29, West 39th Street, New York 18, U.S.A.
Metal Industr.	Metal Industry, Iliffe & Sons Ltd., Dorset House, Stamford Street, London, S.E.1, England.
Mech. World	Mechanical World, Kennedy Press Limited, 31, King Street West, West Manchester 3, England.
Mater. & Meth.	Material and Methods, 330, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Finish.	Metal Finishing, Finishing Publications Inc., 11, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Progr.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Metallurgia	Metallurgia, The Kennedy Press Ltd., 31, King Street West, Manchester 3, England.
Mod. Refrig.	Modern Refrigeration, Refrigeration House, Victoria Road, Woking, Surrey, England.
Murex Rev.	Murex Review, Murex Ltd., Rainham, Essex. England.
Nature	Nature, Macmillan and Co., St. Martin's Street, London, W.C.2, England.
Naturwiss.	Naturwissenschaften, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Nucleonics	Nucleonics, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Oyo Butsuri	Journal of Applied Physics, The Society of Applied Physics, Faculty of Technology, University of Tokio, Japan.
Philips Tech. Rev.	Philips Technical Review, Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2, England.
Philos. Mag.	Philosophical Magazine, Taylor & Francis Limited, 18, Red Lion Court, Fleet Street, London, E.C.4, England.
Physica.	Physica, Martinus Nijhoff, The Hague, Holland.
Phys. Rev.	Physical Review, American Institute of Physics, Brookhaven National Laboratory, Upton, Long Island, N.Y., U.S.A.
Power	Power, McGraw-Hill Publishing Co. Inc., 330, West 42nd St., New York 36, N.Y., U.S.A.
Proc. Inst. Electr. Engrs.	Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, Strand, London, W.C.2, England.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Royal Society, The Royal Society, Burlington House, Piccadilly, London, W.1, England.
Proc. Roy. Soc. Med.	Proceedings of Royal Society of Medicine, 1, Wimpole Street, London, W.1, England.

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Proc. Soc. Exp. Biol. Med.	Proceedings of the Society for Experimental Biology and Medicine, College of the City of New York, New York, 31, N.Y., U.S.A.
Prod. Engng.	Product. Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Prod. Finish.	Product Finishing, Sawell Publication Limited, 4, Ludgate Circus, London, E.C.4, England.
Reader's Dig.	Reader's Digest Association Ltd., 27, Albemarle Street, London, W.1, England.
Refrig. Engng.	Refrigerating Engineering, American Society of Refrigerating Engineers, 40 W. 40th Street, New York 18, N.Y., U.S.A.
Rev. Mod. Phys.	Review of Modern Physics, American Institute of Physics, Lancaster, Pa., U.S.A.
Rev. Prat. Froid	Revue Pratique du Froid, 254, Rue de Vaugirard, Paris 15e, France.
Rev. Pure Appl. Chem.	Review of Pure and Applied Chemistry, The Royal Australian Chemical Institute, Kelvin Hall, 55, Collins Place, Melbourne, Australia.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
Sci. J. Roy. Coll. Sci.	Scientific Journal of the Royal College of Science, Royal College of Science, Imperial Institute Road, London, S.W.7, England.
Science	Science, American Association for the Advancement of Science, 1515, Massachusetts Avenue, N.W., Washington 5, D.C., U.S.A.
Silikattech.	Silikattechnik, Verlag Technik G.m.b.H., Berlin, C, Oranienburger Strasse 13-14.
Stahl u. Eisen	Stahl und Eisen, Düsseldorf, Germany.
Stain Technol.	Stain Technology, Biotech Publications, Geneva, N.Y., U.S.A.
Steel	Steel, Penton Publishing Co., Penton Building, Cleveland 13, Ohio, U.S.A.
Suff. Tech. Pap.	Technical Paper, Suffield Experimental Station, Ralston, Alberta, Canada.
Sylvan. Tech.	Sylvania Technologist, Sylvania Electric Products, Inc., Sylvania Center, Bayside, N.Y., U.S.A.
Times Sci. Rev.	Times Science Review, The Times, London, E.C.4, England.
Trans. Am. Soc. Metals	Transactions of the American Society of Metals, American Society of Metals. 7301, Uclid Avenue, Cleveland 3, Ohio, U.S.A.
Trans. I. Chem. E.	Transactions of the Institution of Chemical Engineers, The Institution of Chemical Engineers, 56, Victoria Street, London, S.W.1, England.
Trans. Faraday Soc.	Transactions of the Faraday Society, The Faraday Society, Aberdeen University Press Limited, 6, Upper Kirkgate, Aberdeen, Scotland.
Univ. Calif. Rad. Lab. Rep.	University of California Radiation Laboratory Reports, University of California, Berkeley, California, U.S.A.
Vakuum-Tech.	Vakuum-Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Le Vide	Le Vide, Société Française des Ingénieurs des Techniciens du Vide, 44, Rue de Rennes, Paris 6e., France.
World Refrig.	World Refrigeration, 181, Kensington High Street, London, W.8, England.
Zavodskaya Labor.	Zavodskaya Laboratoria, g-43, 2-oj Obydenski per.d.14, Moscow, Russia.
Zentralblatt	Zentralblatt für Bakteriologie, Parasitenkunde, Infektionskrankheiten und Hygiene, VEB Gustav Fischer Verlag, Jena, Germany.
Z. Phys.	Zeitschrift für Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.

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